

## EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	17	(("5514122") or ("6623727") or ("6528575") or ("6946518") or ("4395524") or ("7112652") or ("4739008") or ("5942120")).PN.	USPAT; EPO; DERWENT	OR	OFF	2006/11/26 15:12
L2	19	("3215486"   "3231530"   "3341627"   "3717689"   "3853601"   "4242159"   "4260652"   "4268641"   "4277344"   "4280970"   "4388189"   "4976897"   "5266391"   "5525236").PN. OR ("5942120").URPN.	US-PGPUB; USPAT; USOCR	AND	ON	2006/11/26 15:35

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**\*\*See application file for complete search history\*\***

**REF-CITED:**

**U.S. PATENT DOCUMENTS**

<b>PAT-NO</b>	<b>ISSUE-DATE</b>	<b>PATENTEE-NAME</b>	<b>US-CL</b>
<u>3373056</u>	March 1968	Martin	N/A 210/639 N/A
<u>3462362</u>	August 1969	Kollsman	N/A 210/639 N/A
<u>3503789</u>	March 1970	Johnson et al.	N/A 210/500.37 N/A
<u>4230463</u>	October 1980	Henis et al.	N/A 55/68 N/A
<u>4604204</u>	August 1986	Linder et al.	210/490 N/A N/A
<u>4612118</u>	September 1986	Kamiyama et al.	N/A 210/500.37 N/A
<u>4659474</u>	April 1987	Perry et al.	N/A 210/639 N/A

**ART-UNIT:** 136

**PRIMARY-EXAMINER:** Spear; Frank

**ATTY-AGENT-FIRM:** Lackenbach Siegel Marzullo & Aronson

**ABSTRACT:**

Dynamic membranes are obtained by coating on a porous polymeric substrate containing functional groups a chemically reactive hydrophilic polymer from a dilute aqueous solution under pressure and crosslinking said polymer present on the porous substrate as thin layer with low molecular polyfunctional compounds. The new membranes show good solvent and compaction resistance and resistance to separation of the individual layers. They can be used in ultrafiltration and reverse osmosis processes.

32 Claims, 0 Drawing figures

Exemplary Claim Number: 1,7

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**Brief Summary Text - BSTX (45):**

Suitable co-monomers which can be copolymerized with acrylonitrile are monomers which contain, for example, hydrophobic, hydrophilic, polar or ionic groups, especially, for example, vinyl esters having 2 to 18 carbon atoms in the acid moiety, especially vinyl acetate, vinyl **ethers** having 3 to 12 carbon atoms, vinyldipyridine, vinyl chloride, styrene, butadiene, acrylic acid or methacrylic acid or (meth)acrylates, for example those having 1 to 4 carbon atoms in the ester moiety. Further suitable monomers are maleic anhydride, 2-aminoethyl methacrylate and allyl compounds, for example allyl alcohol, allyl- or methallyl-sulfonic acid and their salts (alkali metal salts), allyl halides or methallyl halides, allylamines or allyl p-toluenesulfonates. Further suitable compounds are terpolymers, for example of acrylonitrile, styrene and butadiene (ABS polymers), acrylonitrile/vinyl acetate/methylmethacrylate or acrylonitrile/methyl methacrylate/sodiumallylsulfonate, or tetrapolymers based on acrylonitrile. Component (a) can also contain mixtures of the (co)polymers mentioned.

**Brief Summary Text - BSTX (64):**

(J) Free or etherified N-methylolureas of N-methylolmelamines, for example N,N-dimethylolurea, N,N-dimethylolurea dimethyl **ether**, N,N'-dimethylethylene- or -

propylene-urea, 4,5-dihydroxy-N,N'-di-methylol-ethylene-urea or 4,5-dihydroxy-N,N'-di-methylol-ethyleneurea dimethyl ether and di- to -hexamethylolmelamine, trimethylolmelamine dimethyl ether, pentamethylolmelamine di- to -trimethyl ether and hexamethylol-melamine pentamethyl or hexamethyl ether;

#### **Brief Summary Text - BSTX (65):**

(K) Condensation products of dialkylalkanes containing at least one phenolic hydroxyl group and halogenohydrins, for example the diepoxye obtained from 2,2-bis-(4'-hydroxyphenyl)-propane and epichlorohydrin, as well as glycerol triglycidyl ethers and also corresponding diaziridines;

#### **Brief Summary Text - BSTX (68):**

(N) Further reactive compounds, such as trisacryloyl-hexahydro-s-triazine, epoxides or aziridines. Hydrophilic polymers are used in dilute solutions during the concentration polarization step to react with and to coat the membrane substrate, and, optionally, to modify the support prior to this step. The preferred polymers are polyfunctional polymers which contain aliphatic (acyclic or cyclic), aromatic, or heterocyclic amino groups which can be primary, secondary, or tertiary. Or, alternatively, but less preferred, they may be polymers of hydroxyl or thio-functions. Examples of such polymers are polyethyleneimine (M.W. 150-2,000,000) which can be partially alkylated (methyl iodide) or otherwise modified, polyvinylamine (M.W. 1000 to 2,000,000), polyvinyl alcohol (M.W. 2,000 to 200,000) or partially esterified polyvinyl alcohol, cellulose derivatives, such as ethyl cellulose, carboxymethyl cellulose, hydroxymethylcellulose or hydroxyethyl cellulose, polyvinylaniline, polybenzylamines, polyvinyl mercaptan, polymers of 2-hydroxylethyl or 2-aminoethylmethacrylates, polyvinylimidazoline, amine modified polyepihalohydrin (described in U.S. Pat. No. 1,588,807) polydiallylamine derivatives and polymers containing piperidine rings (described in GB 2,027,614A), condensation products of dicyandiamide, amine (ammonium) salts (NH<sub>2</sub>.sub.4 Cl) and formaldehyde, (US 3 290 310), diamino condensation products of polyanhydrides, aminoalkyl polysulphones, aminoalkyl polyarylene (phenylene) oxides (e.g. amino methylated polyphenylene oxide) and hydrophilic amines containing polymers described in EP 8,945 or polyamido-polyamine epichlorohydrin resins. The above polymers may be in part a copolymer or a polymer containing other monomeric units, block polymers or graft polymers. If they are copolymers, the other monomeric units may or may not contain ionic groups (--SO<sub>3</sub>.sub.2.sup..crclbar., --COO.sup..crclbar., --N.sup..sym.R.sub.3). Examples of such copolymers are such of styrene sulfonate (sodium salt) /vinyl aniline, 2-aminoethylmethacrylate/acrylic acid, vinyl aniline/vinylbenzyltrimethyl ammoniumchloride or vinylamine/vinylsulfonate.

#### **Current US Cross Reference Classification - CCXR (1):**

**210/500.37**

**Current US Cross Reference Classification - CCXR (2):**

**210/500.38**

## Examples 2 to 6

The membranes were obtained from polymer solutions differing in composition in analogy to the production instructions from

5 Example 1. The weight loss of membranes in the following examples was determined in analogy to example 1.

10	Ex- am- ple	B poly- sulfone [wt. %]	C PVP K30 [wt. %]	C PVP K90 [wt. %]	Copolymer A [wt. %] Composition VP/SMA 70:30	Weight loss [%]		
						0.5 h	20 h	36 h
	1	64	28	4	4	14.7	22.6	24.5
	2	64	28	0	8	11.8	21.4	23.1
15	3	64	28	8	0	11.4	26.1	28.3
	4	53.3	33.3	6.6	6.6	21.2	35.2	35.9
	5	53.3	33.3	0	13.3	17.9	34.4	35.3
	6	53.3	33.3	13.3	0	27.2	43.4	44.4

20 The percentages by weight indicated in the table refer to the theoretical content which is to be expected in the membrane in its dry state.

## Examples 7 to 12

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The membranes obtained in accordance with examples 1 to 6 were subjected to contact angle measurements using a contact angle measuring instrument of the type OCAH200 from Data Physics.

30	Example	Membrane from example	Contact angle (dist. water) [degrees]		
			0.1 s	1 s	10 s
	7	1	72	69	69
	8	2	78	74	74
35	9	3	71	68	68
	10	4	74	72	71
	11	5	71	71	69
40	12	6	75	71	71

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L9: Entry 1 of 16

File: USPT

Mar 7, 2006

DOCUMENT-IDENTIFIER: US 7008990 B2  
TITLE: Use of polymeric reaction product

## PRIOR-PUBLICATION:

DOC-ID DATE  
US 20030170306 A1 September 11, 2003

Brief Summary Text (5):

A free radical chain polymerization or copolymerization with an  $\omega$ -unsaturated oligo(methyl methacrylate) with ethyl acrylate, styrene, methyl methacrylate, acrylonitrile and vinyl acetate as copolymers is described in a scientific article in J. Macromol. Sci. Chem. A 23 (7) (1986), 839 852.

Brief Summary Text (8):

Furthermore, Harwood et al. in Macromol. Symp. 111 (1996), 25 35 report on NMR investigations into random, block and graft copolymers using NMR-sensitive initiators and macroinitiators. Inter alia, the reaction of a methyl methacrylate/stilbene mixture and the properties of the polymer resulting therefrom are described there.

Brief Summary Text (11):

In the context of the present invention, a block copolymer is understood as meaning a polymer which has at least two polymer blocks characterized by different monomer compositions. In the context of the present invention, different monomer compositions is understood as meaning that at least two regions of the block copolymer have different monomer compositions. In the context of the present invention, it is possible for the transition between two blocks to be continuous, i.e., for there to exist between two blocks a zone which has a random or regular sequence of the monomers constituting the blocks. In the context of the present invention, however, it is also envisaged that the transition between two blocks is essentially discontinuous. An essentially discontinuous transition is understood as meaning a transition zone which has a substantially shorter length than at least one of the blocks separated by the transition zone. It is possible for a block to be based only on one type of monomer. However, it is also envisaged that a block is composed of two or more monomers. In a preferred embodiment of the present invention, the chain length of such a transition zone is less than 1/10, preferably less than 1/20, of the block length of at least one of the blocks separated by the transition zone.

Brief Summary Text (12):

In the context of the present invention different monomer compositions is furthermore understood as meaning that the monomers constituting the respective block differ in at least one feature, for example in their linkage to one another, in their conformation or in their constitution. If, as described above, a block is composed of more than one type of monomer, in the present context different blocks of the block copolymer can, for example, also differ by having different concentrations of the monomers constituting a block in each case. In the context of the present invention, block copolymers preferably used are those which have at least two blocks whose monomer compositions differ at least in the constitution of

the monomers.

Brief Summary Text (18):

Specific examples of monomers (a) are: Dienes, such as butadiene, isoprene, myrcene, pentadienes, and furthermore C.sub.1- to C.sub.20-alkyl and hydroxyalkyl esters of monoethylenically unsaturated C.sub.3- to C.sub.10-monocarboxylic acids or C.sub.4- to C.sub.8-dicarboxylic acids, for example methyl methacrylate, ethyl methacrylate, propyl methacrylate (all isomers), butyl methacrylate (all isomers), 2-ethylhexyl methacrylate, isobornyl methacrylate, methyl acrylate, ethyl acrylate, propyl acrylate (all isomers), butyl acrylate (all isomers), 2-ethylhexyl acrylate, isobornyl acrylate, benzyl acrylate, phenyl acrylate, stearyl acrylate, diethyl maleate, hydroxyethyl acrylate, hydroxypropyl acrylate, hydroxybutyl acrylate, furthermore (meth)acrylates of alkoxylated C.sub.1- to C.sub.18-alcohols which have been reacted with from 2 to 50 mol of ethylene oxide, propylene oxide, butylene oxide and mixtures thereof; benzyl methacrylate, phenyl methacrylate, stearyl methacrylate, methacrylonitrile, styrene, .alpha.-methylstyrene, acrylonitrile, functionalized methacrylates; acrylates and styrenes selected from glycidyl methacrylate, 2-hydroxyethyl methacrylate, hydroxypropyl methacrylate (all isomers), hydroxybutyl methacrylate (all isomers), cyclohexyl methacrylate, cyclohexyl acrylate, hexyl methacrylate and hexyl acrylate (in each case all isomers), diethylaminoethyl methacrylate, triethylene glycol methacrylate, itaconic anhydride, itaconic acid, glycidyl acrylate, 2-hydroxyethyl methacrylate, diethylaminoethyl acrylate, triethylene glycol acrylate, methacrylamide, N-tert-butylmethacrylamide, N-n-butylmethacrylamide, N-methylolmethacrylamide, N-ethylolmethacrylamide, N-tert-butylacrylamide, N-butylacrylamide, N-methylolacrylamide, N-ethylolacrylamide, vinylbenzoic acid (all isomers), diethylaminostyrene (all isomers), .alpha.-methylvinylbenzoic acid (all isomers), diethylamino-.alpha.-methylstyrene (all isomers), p-methylstyrene, p-vinylbenzenesulfonic acid, trimethoxysilylpropyl methacrylate, triethoxysilylpropyl methacrylate, tributoxysilylpropyl methacrylate, triethoxy-methylsilylpropyl methacrylate, dibutoxymethylsilylpropyl methacrylate, diisopropoxymethylsilylpropyl methacrylate, dimethoxysilylpropyl methacrylate, diethoxysilylpropyl methacrylate, dibutoxysilylpropyl methacrylate, diisopropoxysilylpropyl methacrylate, trimethoxysilylpropyl acrylate, triethoxysilylpropyl acrylate, tributoxysilylpropyl acrylate, dimethoxymethylsilylpropyl acrylate, diethoxymethylsilylpropyl acrylate, dibutoxymethylsilylpropyl acrylate, diisopropoxymethylsilylpropyl acrylate, dimethoxysilylpropyl acrylate, diethoxysilylpropyl acrylate, dibutoxysilylpropyl acrylate, diisopropoxysilylpropyl acrylate, vinyl acetate and vinyl butyrate, vinyl chloride, vinyl fluoride, vinyl bromide, vinyl alcohol, vinyl ethers of C.sub.1- to C.sub.18-alcohols, vinyl ethers of alkoxylated C.sub.1- to C.sub.18-alcohols and vinyl ethers of polyalkylene oxides, such as polyethylene oxide, polypropylene oxide or polybutylene oxide, monoethylenically unsaturated C.sub.3- to C.sub.10-monocarboxylic acids, their alkali metal salts and/or ammonium salts, for example acrylic acid, methacrylic acid, dimethylacrylic acid, ethylacrylic acid, allylacetilic acid or vinylacetic acid, furthermore monoethylenically unsaturated C.sub.4- to C.sub.8-dicarboxylic acids, their monoesters, anhydrides, alkali metal salts and/or ammonium salts, for example maleic acid, fumaric acid, itaconic acid, mesaconic acid, methylenemalonic acid, citraconic acid, maleic anhydride, itaconic anhydride or methylmalonic anhydride; furthermore monoethylenically unsaturated monomers containing sulfo groups and their salts, for example their alkali metal or ammonium salts, for example allylsulfonic acid, styrenesulfonic acid, 2-acrylamido-2-methylpropanesulfonic acid (AMPS), methallylsulfonic acid, vinylsulfonic acid, 3-sulfopropyl acrylate or 3-sulfopropyl methacrylate, furthermore monoethylenically unsaturated monomers containing phosphonic acid groups or their salts, for example their alkali metal or ammonium salts, for example vinylphosphonic acid, allylphosphonic acid or acrylamidoethylpropanephosphonic acid, furthermore amides and N-substituted amides of monoethylenically unsaturated C.sub.3- to C.sub.10-monocarboxylic acids or C.sub.4- to C.sub.8-dicarboxylic acids, for example acrylamide, N-alkylacrylamides or N,N-dialkylacrylamides, each having 1 to 18 carbon atoms in the alkyl group, such as N-methylacrylamide, N,N-

dimethylacrylamide, N-tert-butylacrylamide or N-octadecylacrylamide, N-monomethylhexylmaleamide, N-monodecylmaleamide, diethylaminopropylmethacrylamide or acrylamidoglycollic acid; furthermore alkylaminoalkyl (meth)acrylates, for example dimethylaminoethyl acrylate, dimethylaminoethyl methacrylate, ethylaminoethyl acrylate, diethylaminoethyl methacrylate, dimethylaminopropyl acrylate or dimethylaminopropyl methacrylate; furthermore vinyl esters, such as vinyl formate, vinyl acetate or vinyl propionate, where these may also be present in hydrolyzed form after the polymerization; furthermore N-vinyl compounds, for example N-vinylpyrrolidone, N-vinylcaprolactam, N-vinylformamide, N-vinyl-N-methylformamide, 1-vinyl-imidazole or 1-vinyl-2-methylimidazole; furthermore vinyl ethers of C.sub.1- to C.sub.18-alcohols, vinyl ethers of alkoxylated C.sub.1- to C.sub.18-alcohols and vinyl ethers of polyalkylene oxides, such as polyethylene oxide, polypropylene oxide or polybutylene oxide, styrene or its derivatives such as .alpha.-methylstyrene, indene, dicyclopentadiene, monomers which carry amino or imino groups, such as dimethylaminoethyl methacrylate, diethylaminoethyl acrylate, diethylaminopropyl methacrylamide or allylamine, monomers which carry quaternary ammonium groups, for example present as salts, as obtained by reacting basic amino functions with acids, such as hydrochloric acid, sulfuric acid, nitric acid, formic acid or acetic acid, or in quaternized form (examples of suitable quaternizing agents are dimethyl sulfate, diethyl sulfate, methyl chloride, ethyl chloride or benzyl chloride), e.g. dimethylaminoethyl acrylate hydrochloride, diallyldimethylammonium chloride, dimethylaminoethyl acrylate methylchloride, dimethylaminoethylaminopropylmethacrylamide methosulfate, vinylpyridinium salts or 1-vinylimidazolium salts; monomers in which the amino groups and/or ammonium groups are liberated only after the polymerization and subsequent hydrolysis, for example N-vinylformamide or N-vinylacetamide, and mixtures of two or more of the abovementioned monomers.

Brief Summary Text (31):

However, it is also possible to carry out the reaction according to stage (i) in an organic solvent or in the absence of a solvent, for example in the melt. When the term reaction in an organic solvent or in the absence of a solvent is used in the context of the present invention, it is understood as meaning a reaction which takes place in the presence of less than 10, preferably less than 5 or less than 1, % by weight of water. In a further embodiment of the present invention, at least one block copolymer in whose preparation stage (i) was carried out in an organic solvent or in the absence of a solvent is used in the novel binder composition, the water content of the reaction mixture being less than 0.5, for example less than 0.3 or less than 0.1, % by weight. In a further embodiment of the present invention, the reaction of stage (i) is carried out in the absence of water, i.e. with a water content of less than 0.001% by weight. Such water contents can be achieved, for example, by using commercially available solvents, as usually used as organic solvents in free radical polymerizations.

Brief Summary Text (46):

Depending on the reaction procedure, it is possible according to the invention to prepare polymers functionalized at the terminal groups, segmented polymers, block, multiblock or gradient (co)polymers, star (co)polymers, graft copolymers and branched and hyperbranched (co)polymers.

Brief Summary Text (47):

As is evident from the above, the present invention also relates to the use of the polymer (B), which can be prepared by the process defined above, for numerous applications. The reaction is preferably carried out in such a way that a polymer (B) which has a block structure is obtained. Using an easily obtainable compound (I), it is possible in a simple manner to provide block copolymers which have, for example, a hydrophilic block, e.g. a (meth)acrylic acid or a C.sub.1-4-alkyl (meth)acrylate block, and a fisher, preferably hydrophobic polymer block, e.g. a block based on vinylaromatic monomers, such as styrene or substituted styrenes, acrylonitrile, dienes and nonaromatic vinyl compounds, such as vinyl acetate, and

higher (>C.sub.4) alkyl (meth)acrylates.

Brief Summary Text (49):

Specific examples are the following block copolymers: Poly(acrylic acid-b-styrene), poly(methyl methacrylate-b-styrene), poly(styrene-b-vinyl acetate), poly(methacrylic acid-b-hydroxyethyl acrylate), poly(methyl methacrylate-b-N-vinylpyrrolidone), poly(methyl methacrylate-b-N-vinylformamide), poly(methyl methacrylate-b-hydroxyethyl acrylate), poly(methyl methacrylate-b-(styrene-stat-acrylonitrile)), poly(n-butyl acrylate-b-styrene-b-n-butyl acrylate), poly(methyl methacrylate-b-styrene-b-methyl methacrylate-b-styrene), poly(n-butyl acrylate-b-styrene-b-n-butyl acrylate-b-styrene), poly((meth)acrylic acid-stat-(meth)acrylate-b-(styrene-stat-(meth)acrylate)).

Brief Summary Text (50):

Specific examples are the following block copolymers: Poly(styrene-b-acrylic acid), poly(styrene-b-methyl acrylate), poly(styrene-b-ethyl acrylate), poly(styrene-b-methacrylic acid), poly(styrene-b-methyl methacrylate), poly(styrene-b-ethyl methacrylate), poly(hydroxyethyl acrylate-b-methacrylic acid), poly(N-vinylpyrrolidone-b-methyl acrylate), poly(N-vinylpyrrolidone-b-ethyl acrylate), poly(N-vinylpyrrolidone-b-methyl methacrylate), poly(N-vinylpyrrolidone-b-ethyl methacrylate), poly(N-vinylpyrrolidone-b-styrene), poly(N-vinylpyrrolidone-b-vinyl acetate), poly(N-vinylpyrrolidone-b-(.alpha.-methylstyrene)), poly(N-vinylformamide-b-methyl methacrylate), poly(N-vinylformamide-b-ethyl methacrylate), poly(N-vinylformamide-b-vinyl acetate), poly(N-vinylformamide-b-methyl acrylate) or poly(N-vinylformamide-b-ethyl acrylate).

Description Paragraph (9):

Toner compositions can be prepared by various known methods, for example by mixing together and heating resin particles which contain the components (A) or (B) described herein, for example corresponding styrene/butadiene copolymers, with pigments, such as magnetite, carbon black or mixtures thereof, and colored pigments, such as cyan, magenta, yellow, green, brown or red pigments or mixtures thereof, and preferably from 0.5 to 5% by weight of an additive for increasing the charge in an extrusion apparatus for toners, for example ZSK 53 from Werner & Pfleiderer, and subsequently removing the resulting toner composition from the apparatus. After cooling, the toner composition is milled in a suitable micronizing apparatus in order to obtain toner particles which have a mean diameter of less than about 25 .mu.m, preferably from 6 to 12 .mu.m, these diameters being determined using a Coulter counter. This is followed by classification of the particles, toner particles having a diameter of less than 4 .mu.m being removed.

Description Paragraph (30):

Conventional thickeners in such formulations are crosslinked polyacrylic acid and its derivatives, polysaccharides, such as xanthan gum, agar agar, alginates or tyloses, cellulose derivatives, for example carboxymethylcellulose or hydroxycarboxymethylcellulose, fatty alcohols, monoglycerides and fatty acids, polyvinyl alcohol and polyvinylpyrrolidone.

Description Paragraph (34):

Regarding the use of components (A) and (B) described herein as resin material, reference is made to DE-A 196 36 058, which describes a styrene resin material. It is of course also possible to prepare other resin materials, for example polymers based on acrylic acid/butadiene and acrylic acid/styrene or acrylic acid/styrene/acrylic acid or copolymers of styrene and acrylonitrile in the same or an analogous manner. Such resin materials are described below using a styrene resin material as an example.

Description Paragraph (38):

Before they are processed to give moldings, sheets, fibers and foams, the polymers are generally mixed with additives which are useful for modifying the basic

properties (modifiers, plasticizers, fillers and reinforcing materials, flameproofing agents, antistatic agents, dyes, pigments, etc.) or for carrying out the processing in a trouble-free manner (stabilizers, lubricants, mold release agents, etc.). The novel polymers used can also be employed as a mixture with other polymers, such as polyethylene terephthalate (PET), polybutylene terephthalate (PBT), polycarbonate (PC), polyamide (PA 6), polyamide 66 (PA 66), polyamide 12 (PA 12), polyamide 4,6 (PA 4,6), copolyamides, polypropylene oxide (PPO), polyetherimides, polyetherketones, polyimides, acrylonitrile/butadiene/styrene (polymers) (ABS), acrylonitrile/styrene/acrylate (polymers) (ASA), poly (amidoimides), polybutadiene, poly(meth)acrylate, epoxy resins, polyethylene (PE), polypropylene (PP), EPDM (ethylenepropylenediene monomer rubber), copolymers of .alpha.-olefins, polyvinyl chloride (PVC), polymethyl methacrylate (PMMA), polystyrene (PS), styrene/acrylonitrile copolymers (SAN), polyvinyl alcohol, polyvinyl acetate, thermoplastic polyurethane elastomers (TPU), polylactide, and polymers described in chapter 5 of Polymer Handbook, 3rd ed., Brandrup, J. and Immergut, E H, published by John Wiley & Sons, 1989, New York, and their blends and copolymers and block copolymers. Further suitable additives and polymers are known to those skilled in the art.

Description Paragraph (65):

The components (A) and/or (B) described herein, preferably copolymers of the type described above which have been rendered hydrophobic, can also be used as incrustation inhibitors and/or soil release polymers in detergents. Regarding the general formulation of such detergents and the function as incrustation inhibitor and/or soil release polymer, reference may be made to DE-A 196 08 044.

Description Paragraph (72):

In connection with soil release polymers, it should be mentioned here that the polymers (B) described herein are also particularly suitable owing to the possibilities of modifying them in a virtually freely selectable manner with regard to their properties in the preparation process described here. For example, it is preferable to use in particular amphiphilic graft polymers or copolymers as described herein, amphiphilic graft polymers or copolymers of vinyl esters and/or acrylates on polyalkylene oxides being mentioned in particular.

Description Paragraph (75):

It is particularly advantageous to meter the filtration aid, i.e. the components (A) and (B) described herein, continuously into the unfiltered material. This can be realized in particular by metering in one operation or by metering in the crossflow circulation. It is advantageous if the stabilizer is pumped with the circulation of unfiltered material through the membrane filter of the crossflow filtration unit. The circulation may be closed directly from the outlet of the filtration unit via a pump back to the inlet thereof. However, it is also possible to close the circulation via the working tank, i.e. to pump the unfiltered material continuously from the working tank through the membrane filter and to transport the retentate present at the outlet of the filtration unit, together with the stabilizer contained therein, back into the working tank. Owing to the small pore size of such crossflow membrane filters (from 0.001 to 1 .mu.m in the case of ultrafiltration or from about 0.1 to 1 .mu.m in the case of microfiltration), the entry of bound or precipitated tannins or proteins into the filtrate is reliably prevented. Instead, these are circulated with the retentate on the unfiltered material side of the membrane filter until the end of a filtration cycle.

Description Paragraph (76):

The filtration aid may be metered in without recovery and removed from the filtration unit together with the sediments after the end of a filtration cycle. Polyvinylpolypyrrolidone (PVPP) and further block copolymers described herein and silica gel have proven particularly useful.

Description Paragraph (77):

If the unfiltered material contains large amounts of suspended matter, the filtration performance of the membrane filters can be increased if granular or fibrous filtration aids are fed to the membranes for protecting the membrane pores from blockage by sediments

Description Paragraph (78):

Granular is understood as describing those filtration aids which consist of regularly or irregularly shaped particles which neither dissolve in the unfiltered material nor agglomerate or are otherwise compacted under the influence of the transmembrane pressure. According to the invention, filtration aids consisting of such granular particles are deposited in spite of the crossflow along the membrane surface and can thus prevent the formation of continuous top layers of sediments.

Description Paragraph (79):

The filtration aid can advantageously be applied as a top layer directly on the membrane surface. Consequently, the pores of the membrane surface are reliably protected from blockage by sediments. The top layer should have a layer thickness of 1 to 20 .mu.m, preferably from 1 to 8 .mu.m; it therefore does not constitute a filter layer in the conventional sense but a protective layer for the actual membrane filter layer.

Description Paragraph (80):

The filtration aid can be applied to the membrane surface before the beginning of the filtration process. This produces a top layer which consists exclusively of the filtration aid and is free of contamination by sediments. This can be achieved if first a medium which contains no substantial amounts of suspended matter is caused to flow across the membrane in order to start up a filtration cycle, if filtration aid is added to the medium in order to form the top layer and if the unfiltered material is then fed to the membrane. Such a top layer can be easily produced if the filtration cycle is started up with water to which filtration aid is added. As soon as the top layer has been produced, the water can be forced out of the filter as a forward flow and unfiltered material can be fed in.

Description Paragraph (82):

The protection of the membrane pores from blockage is ensured in a particularly reliable manner if filtration aid having a particle size which is greater than the pore size of the filter membrane is applied. A particle size of from 1 to 80 .mu.m has proven particularly useful, and, by an appropriate choice of the particle size distribution, for example from 60 to 80% of the particles in the range from 1 to 4 .mu.m, it is possible to optimize the filtration aid in this range with respect to the medium to be filtered and the suspended matter contained therein, Particularly good permeability and a large filtration surface can be achieved if kieselguhr is used as filtration aid.

Description Paragraph (85):

Regarding this use, reference may be made herein to EP-A 0 756 820, which relates to the use of dextrins in disinfectants. However, these dextrins alone are not capable of forming sufficiently stable iodine complexes. Surprisingly, however, they can do so when mixed with the reaction products (A) or the polymers (B) described herein, since these copolymers are likewise capable of taking up iodine, and are able to do so with a bond force comparable to that of dextrin, for example homopolyvinylpyrrolidones and block copolymers of N-vinylpyrrolidone and styrene, methyl methacrylate, methyl acrylate, hydroxymethyl acrylate or hydroxyethyl acrylate.

Description Paragraph (92):

The formulations are suitable in particular for use in the coarse disinfection of surfaces as well as fine disinfections. Thus, they can be used in compositions for the antiseptic treatment of skin and mucous membranes or for disinfecting hands for surgical and hygiene purposes.

Description Paragraph (98):

Examples of suitable elastomeric binders are elastomeric polymeric binders, for example polyalkadienes, vinylaromatic/alkadiene copolymers and block polymers, alkadiene/acrylonitrile copolymers, ethylene/propylene copolymers, ethylene/propylene/alkadiene copolymers, ethylene/acrylic acid copolymers, alkadiene/acrylic acid copolymers, alkadiene/acrylate/ acrylic acid copolymers and ethylene/(meth)acrylic acid/(meth)acrylate copolymers, in each case prepared as described above.

Description Paragraph (108):

The cylinder layer crosslinkable by actinic radiation generally has a thickness of from 200 to 8000 .mu.m, in particular from 500 to 6000 .mu.m. A further thin layer which may have a thickness of from 1 to 5 .mu.m and which detackifies the surface of the photosensitive cylinder layer may be applied thereon. Present on the latter or preferably directly on the cylinder layer crosslinkable by actinic radiation is the IR-sensitive layer, which is a layer which is soluble or dispersible in developers and contains, in a film-forming binder having elastomeric character, at least one finely divided substance which has high absorption in the wavelength range from 750 to 20,000 nm and an optical density  $\geq 2.5$  in the actinic range. Developers may be water and water/alcohol or organic solvent (mixtures). Suitable binders having elastomeric character for the IR-sensitive layer are polymers, in particular copolymers, which are either water-soluble or dispersible in water or water/alcohol mixtures or those which are soluble or dispersible in organic solvents or solvent mixtures. Suitable alcohols in the water/alcohol mixtures are methanol, ethanol, n-propanol and isopropanol.

Description Paragraph (109):

Examples of binders which are soluble or dispersible in water or in water/alcohol mixtures and have elastomeric character are polyvinyl alcohol/polyethylene glycol graft copolymers (for example Mowiol.RTM. 597 from Hoechst Aktiengesellschaft, Germany), which are obtainable by grafting vinyl acetate onto polyethylene glycol having molecular weights of from 1000 to 50,000 and then carrying out hydrolysis to a degree of hydrolysis of from 90 to 100%.

Description Paragraph (113):

Synthetic oligomers or resins, such as oligostyrene, oligomeric styrene/butadiene copolymers, oligomeric .alpha.-methylstyrene/p-methylstyrene copolymers, liquid oligobutadienes, liquid oligoisoprenes or liquid oligomeric acrylonitrile/butadiene copolymers, each prepared as described above, may also be used. Such oligomers are molecules having a molecular weight of from 500 to 5000 g/mol.

Description Paragraph (123):

Other suitable thickeners in addition to the novel components (A) and (B) themselves are polymers of hydrophilic monomers capable of free radical polymerization, such as acrylic acid or methacrylic acid, polyvinylpyrrolidone or thickeners based on cellulose or starch derivatives, such as carboxymethylcellulose, carboxyethylcellulose, hydroxyethylcellulose, hydroxypropylcellulose, hydroxyethyl starch, hydroxypropyl starch and the like. Said thickeners may each be present individually or as a mixture of two or more thereof in the novel dispersions.

Description Paragraph (147):

The novel pigment formulations can be printed on all types of substrate materials. Examples of substrate materials which may be mentioned are cellulose-containing materials, such as paper, board, cardboard, wood and woodbase materials, which may also have been treated with a finish or otherwise coated, metallic materials, such as foils, sheets or workpieces made of aluminum, iron, copper, silver, gold, zinc or alloys of these metals, which may have been treated with a finish or otherwise coated, silicate materials, such as glass, porcelain and ceramic, which may

likewise have been coated, polymeric materials of any type, such as polystyrene, polyamides, polyesters, polyethylene, polypropylene, melamine resins, polyacrylates, polyacrylonitrile, polyurethanes, polycarbonates, polyvinyl chloride, polyvinyl alcohols, polyvinyl acetates, polyvinylpyrrolidones and corresponding copolymers and block copolymers, biodegradable polymers and natural polymers, such as gelatin, textile materials, such as fibers, yarns, thread, knits, wovens, nonwovens and ready-made goods composed of polyester, modified polyester, polyester blend fabrics, cellulose-containing materials, such as cotton, cotton blend fabrics, jute, flax, hemp and ramie, viscose, wool, silk, polyamide, polyamide blend fabrics, polyacrylonitrile, triacetate, acetate, polycarbonate, polypropylene, polyvinyl chloride, polyester microfibers and glass fiber fabric, leather, both natural and artificial, in the form of smooth leather, nappa leather or suede leather, food and cosmetics.

Description Paragraph (153):

Possible additional formulation excipients are a) meltable sugar alcohols, sugars, fats and waxes (from 0 to 99%), b) polymers, such as polyvinylpyrrolidone, cellulose derivatives, polyvinylformamide (also partially or completely hydrolyzed), copolymers, polyethylene glycols, starch and starch derivatives, polyacrylates and polymethacrylates (Eudragit types), polyvinyl alcohol, partially hydrolyzed polyvinyl acetate and polyacrylamides (from 0 to 99%), c) if required, assistants such as surfactants, disintegrants, colorants, lubricants or plasticizers, dispersants, fillers or salts and antifoams (from 0 to 99%) or other mixtures.

Description Paragraph (157):

In the preparation of the formulations, it is of course also possible to add further excipients conventionally used in the preparation of solid oral dosage forms. These may be substances from among fillers and binders (for example lactose, calcium phosphates, cellulose and its derivatives, starch, polyvinylpyrrolidone, polyvinylformamide (also partially or completely hydrolyzed), polyvinyl alcohol, partially hydrolyzed polyvinyl acetate, polyacrylamides (from 0 to 99%), sugar alcohols, sugars, fats or waxes (from 0 to 99%)), disintegrants (for example Kollidon C L, according to the claim carboxymethyl starch or carboxymethylcellulose), lubricants (for example magnesium stearate, calcium behenate, stearic acid or PEG), flow regulators (for example finely divided silica), film formers (for example polyacrylates and polymethacrylates (Eudragit types), copolymers based on acrylate derivatives, hydroxypropylmethylcellulose, hydroxypropylcellulose, cellulose acetate, cellulose acetate phthalate and other coating materials resistant to gastric fluid), humectants (for example glycerol, propylene glycol, sorbitol, mannitol or polyethylene glycols), plasticizers, colorants, surfactants, salts and dispersants.

Description Paragraph (160):

The novel components (A) or (B) are extremely useful as additives for cement mixes, such as concrete or mortar. Cement is to be understood as meaning, for example, Portland cement, high-alumina cement or mixed cement, for example pozzolanic cement, slag cement or other types. Portland cement is preferred. The copolymers are used in an amount of from 0.01 to 10, preferably from 0.05 to 3, % by weight based on the weight of the cement.

Description Paragraph (161):

The components (A) and (B) can be added in solid form, which is obtainable by drying, for example spray-drying, of polymer solutions or dispersions as obtained in the polymerization, to the ready-to-use preparation of the mineral building material. It is also conceivable to formulate the copolymers with the mineral binder and to prepare the ready-to-use preparation of the mineral building materials therefrom. Preferably, the copolymer is used in liquid, i.e. dissolved, emulsified or suspended, form, for example in the form of the polymerization solution, in the preparation of the mineral building material.

Description Paragraph (164):

In principle, the novel components (A) and (B) can also be used together with film-forming polymers. These are understood as meaning those polymers whose glass transition temperature (DSC midpoint temperature, ASTM D 3481 82) is  $\geq 65$  degree C., preferably  $\geq 50$  degree C., particularly preferably 25 degree C., very particularly preferably  $\geq 0$  degree C. On the basis of the relationship between the glass transition temperature of homopolymers and the glass transition temperature of copolymers, postulated by Fox (T. G. Fox, Bull. Am. Phys. Soc. (Ser. II) 1 (1956), 123), those skilled in the art are able to select suitable polymers (glass transition temperatures for homopolymers are to be found, for example, in Ullmanns Encyclopedia of Industrial Chemistry, Vol. A21, VCH, Weinheim 1992, page 169, or in J. Brandrup, E. H. Immergut, Polymer Handbook, 3.sup.rd Ed., J. Wiley, New York 1998).

Description Paragraph (180):

Suitable polymer compounds redispersible in water are the reaction products (A) and/or polymers (B) described herein. Both homopolymers and/or copolymers of the classes of substances selected in each case are suitable here. Typical members of these polymer compounds are vinyl esters of lower carboxylic acids, vinyl acetate and/or vinyl propionate being particularly important. In addition to the is homopolymers of this type, copolymers, for example vinyl acetate/maleate copolymers or ethylene/vinyl acetate copolymers, are used in practice. A further important class comprises corresponding (meth)acrylate homo- and/or copolymers, an example of suitable copolymers being styrene acrylate.

Description Paragraph (197):

Examples of aqueous systems which can be thickened according to the invention are aqueous polyacrylate dispersions, aqueous dispersions of copolymers of olefinically unsaturated monomers, aqueous polyvinyl acetate dispersions, aqueous polyurethane dispersions, aqueous polyester dispersions and in particular ready-to-use formulations of the type discussed above and based on such dispersions.

Description Paragraph (202):

The components (A) and (B) described herein can also be used as components in pressure-sensitive adhesive materials. Such components (A) and/or (B) based on block copolymers containing polymer blocks formed from vinylaromatics (A blocks), preferably styrene, and those formed by polymerization of 1,3-dienes (D blocks), preferably butadiene and isoprene, are preferably employed.

Description Paragraph (203):

According to the invention, both homopolymer and copolymer blocks can be used. The resulting block copolymers may contain identical or different D blocks, some or all of which can be completely hydrogenated or which can be selectively hydrogenated. Block copolymers may have a linear A-D-A structure. Block copolymers having a radial structure and star and linear multiblock copolymers may also be used. A-D two-block copolymers may be present as further components. Block copolymers may be modified, for example functionalized by reaction with maleic anhydride. According to the invention, block copolymers of vinylaromatics and isobutylene can also be used. All of the abovementioned polymers can be used alone or in mixture with one another. Typical concentrations in which the styrene block copolymers are used are from 15 to 75, preferably from 30 to 60, particularly preferably from 35 to 55, % by weight.

Description Paragraph (205):

Homopolymers and copolymers of vinylaromatics, for example styrene or  $\alpha$ -methylstyrene, polyphenylene oxides and also phenylene oxide-modified resins may be used as resins compatible with the terminal blocks, chiefly resins compatible with the vinylaromatic blocks.

Description Paragraph (206):

Further optimum components of the mixture comprise plasticizer oils and liquid resins (concentration of use from 0 to not more than about 35% by weight), fillers (reinforcing and nonreinforcing), for example silica, in particular synthetic silica, glass (milled or in the form of beads), aluminas, zinc oxides, calcium carbonates, titanium dioxide, carbon blacks, to mention but a few, and antiaging compositions (primary and secondary antioxidants, light stabilizers, antiozonants, metal deactivators, etc.). Components of the mixture also comprise polymers which in particular affect the ozone resistance of the block copolymers, for example polyvinyl acetates and ethylene/vinyl acetate copolymers.

Description Paragraph (207):

Further polymers which may be used are natural and synthetic polymers, for example natural rubber, synthetic polyisoprenes, polybutadienes, polychloroprenes. SBR, Kraton Liquid (Shell Chemicals), low molecular weight styrene/diene block copolymers, for example Kraton LVSI 101, polyisobutylene, etc., which may replace up to about 50% by weight of the vinylaromatic-containing block copolymers.

Description Paragraph (210):

Suitable pressure-sensitive adhesive materials in addition to those described above and based on vinylaromatic-containing block copolymers are all those which have tensile strength and cohesion sufficient for the release process. Corresponding pressure-sensitive adhesive materials can be used alone or in combination with those based on vinylaromatic-containing block copolymers. For example, tacky acrylate copolymers copolymerized with macromonomers are suitable according to the invention, the macromonomers having a glass transition temperature of >+40.degree. C. The high tensile strength of corresponding copolymers is probably achieved by the association of the macromonomers. Suitable macromonomers are, for example, methacryloyl-terminated polymethyl methacrylates,

Description Paragraph (214):

According to the present inventor, the components (A) and (B) can also be used in solid adhesives in order to impart to them the adhesion which they produce. Particularly used components (A) and (B) are those which may be selected from the group consisting of polyvinyl acetate homopolymers, polyvinyl acetate copolymers, partially or completely hydrolyzed polyvinyl alcohol, polyvinyl butyral, polyvinylpyrrolidone, polyacrylic acid salts, polymethacrylic acid salts, polyacrylate, polymethacrylate, various gums, polysaccharides and rubber. The components (A) and/or (B) may be a mixture of more than one of these chemicals. Preferably, the components (A) and/or (B) are a mixture of polyvinyl alcohol and polyvinylpyrrolidone. It has been found that at least about 15%, preferably at least 20%, of the components (A) and/or (B) in the solid adhesive result in good adhesion by the solid adhesive. Too large an amount of the components (A) and/or (B) cannot be dissolved in water. The components (A) and/or (B) are therefore preferably limited to from about 15 to 42%, better still from about 20 to 36%, in the solid adhesive.

Description Paragraph (223):

The novel components (A) and (B), in particular the novel block copolymers, are extremely useful for producing self-cleaning surfaces of articles, as described, for example, in WO 96/04123, the relevant content of which is hereby fully incorporated by reference in the context of the present application. Particularly advantageous block copolymers are those which, owing to the incompatibility of their respective blocks form self-cleaning (i.e. hydrophobic and correspondingly structured) surfaces by self-organization after application.

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1. Document ID: US 7008990 B2

L9: Entry 1 of 16

File: USPT

Mar 7, 2006

US-PAT-NO: 7008990

DOCUMENT-IDENTIFIER: US 7008990 B2

TITLE: Use of polymeric reaction product

DATE-ISSUED: March 7, 2006

PRIOR-PUBLICATION:

DOC-ID	DATE
US 20030170306 A1	September 11, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Raether; Roman Benedikt	Limburgerhof			DE
Brinkmann-Rengel; Susanne	Ober-Olm			DE
Haremza; Sylke	Neckargemund			DE

US-CL-CURRENT: 524/457; 524/458, 525/64, 526/204

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2. Document ID: US 6936394 B2

L9: Entry 2 of 16

File: USPT

Aug 30, 2005

US-PAT-NO: 6936394

DOCUMENT-IDENTIFIER: US 6936394 B2

\*\* See image for Certificate of Correction \*\*

TITLE: Replenishing developer and developing method

DATE-ISSUED: August 30, 2005

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Okado; Kenji	Ushiku			JP
Mikuriya; Yushi	Numazu			JP
Yoshizaki; Kazumi	Toride			JP

Ikeda; Naotaka

Toride

JP

US-CL-CURRENT: 430/111.33; 399/255, 399/259, 430/108.1, 430/108.8, 430/111.1,  
430/111.3, 430/111.31, 430/111.35

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3. Document ID: US 6919073 B2

L9: Entry 3 of 16

File: USPT

Jul 19, 2005

US-PAT-NO: 6919073

DOCUMENT-IDENTIFIER: US 6919073 B2

TITLE: Pulverulent composition for bleaching human keratin fibers

DATE-ISSUED: July 19, 2005

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Legrand; Frederic	Courbevoie			FR
Millequant; Jean-Marie	Saint-Maur des Fosses			FR

US-CL-CURRENT: 424/62; 132/208, 424/701

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [KOMC](#) | [Drawn D](#)

4. Document ID: US 6838078 B2

L9: Entry 4 of 16

File: USPT

Jan 4, 2005

US-PAT-NO: 6838078

DOCUMENT-IDENTIFIER: US 6838078 B2

TITLE: Film-forming compositions and methods

DATE-ISSUED: January 4, 2005

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wang; Danli	Shoreview	MN		
Scholz; Matthew T.	Woodbury	MN		
Zhu; Dong-Wei	Woodbury	MN		
Lu; Triet M.	Woodbury	MN		

US-CL-CURRENT: 424/78.02; 424/400, 424/401, 424/404, 424/405, 424/78.08, 424/78.18,  
424/78.19

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [KOMC](#) | [Drawn D](#)

5. Document ID: US 6833419 B2

L9: Entry 5 of 16

File: USPT

Dec 21, 2004

US-PAT-NO: 6833419

DOCUMENT-IDENTIFIER: US 6833419 B2

TITLE: Fluorine-modified comb polymers based on acryloydimethyltaurine acid

DATE-ISSUED: December 21, 2004

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Morschhauser; Roman	Mainz			DE
Kayser; Christoph	Mainz			DE
Loffler; Matthias	Niedernhausen			DE

US-CL-CURRENT: 526/288; 526/250, 526/277, 526/303.1, 526/307.1, 526/307.2[Full](#) [Title](#) [Citation](#) [Front](#) [Review](#) [Classification](#) [Date](#) [Reference](#) [Specification](#) [Drawings](#) [Claims](#) [KWMC](#) [Drawn D](#) 6. Document ID: US 6528073 B2

L9: Entry 6 of 16

File: USPT

Mar 4, 2003

US-PAT-NO: 6528073

DOCUMENT-IDENTIFIER: US 6528073 B2

\*\* See image for Certificate of Correction \*\*

TITLE: Solid cosmetic composition and uses thereof

DATE-ISSUED: March 4, 2003

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Roulier; Veronique	Paris			FR
Quemin; Eric	Tremblay en France			FR

US-CL-CURRENT: 424/401, 424/400, 424/449, 424/484, 424/485, 424/486, 424/487,  
424/488, 424/62, 424/63, 424/64, 424/65, 424/70.1, 424/70.11, 424/70.13, 424/70.15,  
424/70.16, 424/70.6, 424/70.7, 514/844, 514/944[Full](#) [Title](#) [Citation](#) [Front](#) [Review](#) [Classification](#) [Date](#) [Reference](#) [Specification](#) [Drawings](#) [Claims](#) [KWMC](#) [Drawn D](#) 7. Document ID: US 6280750 B1

L9: Entry 7 of 16

File: USPT

Aug 28, 2001

US-PAT-NO: 6280750

DOCUMENT-IDENTIFIER: US 6280750 B1

TITLE: Solid cosmetic composition and uses thereof

DATE-ISSUED: August 28, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Roulier; Veronique	Paris			FR
Quemin; Eric	Tremblay en France			FR

US-CL-CURRENT: 424/401, 424/400, 424/484, 424/485, 424/486, 424/487, 424/488,  
424/63, 424/64, 424/65, 424/70.13, 424/70.7, 424/DIG.5, 426/573, 514/944

[Full](#) [Title](#) [Citation](#) [Front](#) [Review](#) [Classification](#) [Date](#) [Reference](#) [Detailed Description](#) [Abstract](#) [Claims](#) [KMC](#) [Drawn D](#)

8. Document ID: US 6214936 B1

L9: Entry 8 of 16

File: USPT

Apr 10, 2001

US-PAT-NO: 6214936

DOCUMENT-IDENTIFIER: US 6214936 B1

\*\* See image for Certificate of Correction \*\*

TITLE: Use of microphase-separated polymer blends for the preparation of permeable membranes

DATE-ISSUED: April 10, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mehler; Christof	67061 Ludwigshafen			DE
Gottschalk; Axel	67435 Neustadt			DE
Breiner; Ulrike	68642 Burstadt			DE
Stadler; Reimund Erich	late of Glashutten			DE
Widmer-Stadler; Cacilda Pereira	95496 Glashutten			DE
Goldacker; Thorsten	95445 Bayreuth			DE

US-CL-CURRENT: 525/89, 523/106, 524/505, 525/316, 525/94

[Full](#) [Title](#) [Citation](#) [Front](#) [Review](#) [Classification](#) [Date](#) [Reference](#) [Detailed Description](#) [Abstract](#) [Claims](#) [KMC](#) [Drawn D](#)

9. Document ID: US 6165663 A

L9: Entry 9 of 16

File: USPT

Dec 26, 2000

US-PAT-NO: 6165663

DOCUMENT-IDENTIFIER: US 6165663 A

\*\* See image for Certificate of Correction \*\*

TITLE: Magnetic coated carrier two-component type developer and developing method

DATE-ISSUED: December 26, 2000

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Baba; Yoshinobu	Yokohama			JP
Ikeda; Takeshi	Shizuoka-ken			JP
Sato; Yuko	Numazu			JP
Itabashi; Hitoshi	Yokohama			JP
Tokunaga; Yuzo	Yokohama			JP

US-CL-CURRENT: 430/111.3; 430/111.35, 430/111.41, 430/122
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 10. Document ID: US 6066316 A

L9: Entry 10 of 16

File: USPT

May 23, 2000

US-PAT-NO: 6066316

DOCUMENT-IDENTIFIER: US 6066316 A

TITLE: Fine dispersion composition of wax, hair cosmetic preparation and glazing agent

DATE-ISSUED: May 23, 2000

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Shiojima; Yoshihiro	Kanagawa			JP
Omura; Takayuki	Kanagawa			JP
Nakama; Yasunari	Kanagawa			JP
Harusawa; Fuminori	Kanagawa			JP

US-CL-CURRENT: 424/70.19; 424/401, 424/70.1, 424/70.21, 424/70.31, 514/937, 514/938
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Terms	Documents
L8 and membrane	16

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<b>Term:</b>	<input type="text" value="L8 and membrane"/> <div style="display: flex; justify-content: flex-end; margin-top: -10px;"> <span style="border: 1px solid black; padding: 2px 5px; margin-right: 10px;">Up</span> <span style="border: 1px solid black; padding: 2px 5px; margin-right: 10px;">Down</span> </div>
<b>Display:</b>	<input type="text" value="10"/> Documents in <b>Display Format:</b> <input type="text" value="CIT"/> Starting with Number <input type="text" value="1"/>
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<u>L9</u>	L8 and membrane	16	<u>L9</u>
<u>L8</u>	L7 and copolymer	112	<u>L8</u>
<u>L7</u>	polyvinylpyrrolidone and stearyl acrylate	118	<u>L7</u>
<u>L6</u>	copolymer same polyvinylpirrolidone same stearyl methacrylate	0	<u>L6</u>
<u>L5</u>	copolymer and vp:stma	0	<u>L5</u>
<u>L4</u>	L3 and copolymer same vinylpyrrolidone and stearil methacrylate	0	<u>L4</u>
<u>L3</u>	polysulfone and n-vinylactam	26	<u>L3</u>
<u>L2</u>	membrane and hydrophobic polymer and copolymer same n-vinylpirrolidone	0	<u>L2</u>
<u>L1</u>	"ep 0953358"	0	<u>L1</u>

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L18: Entry 1 of 1

File: USPT

May 22, 2001

DOCUMENT-IDENTIFIER: US 6235835 B1

\*\* See image for Certificate of Correction \*\*

TITLE: Polymer-modified anionic starch, method for its production, and its use

Brief Summary Text (9):

DE-A-37 19 480 and EP-A-0 282 761 describe a process for the production of paper, board and cardboard having high dry strength by the addition of a dry strength agent comprising a mixture of cationic polymers and starch to the paper stock. The cationic polymers contain, as characteristic monomers, polymerized units of diallyldimethylammonium chloride, N-vinylamine or N-vinylimidazoline. Polyethylene imine may also be used as the cationic polymer. For the preparation of the dry strength agents, either an aqueous suspension of a natural potato starch is digested in the presence of the polymers by heating to above the glutinization temperature in the absence of oxidizing agents, polymerization initiators and alkalis or a digested potato starch is reacted with the cationic polymers at from 15 to 70.degree. C. Exclusively natural or thermally degraded potato starch is used and the modification with the cationic polymers is carried out in aqueous suspension or in aqueous solution.

Brief Summary Text (29):

During digestion, the starch is preferably dissolved to give a clear solution so that, after the reaction, no more unconverted starch can be filtered off during filtration of the reaction solution with a cellulose acetate membrane having a pore diameter of 1.2 .mu.m. If desired, however, any insoluble starch residues present can be separated from the product in this manner. The degree of degradation of the starch can be quantitatively determined with the aid of gel permeation chromatography. The degree of starch digestion from the swollen starch grain to the completely dissolved starch can be determined with the aid of investigations by microscopy and electron microscopy.

Brief Summary Text (35):

a) N-vinylamines of Formula I ##STR1##

Brief Summary Text (61):

In a preferred embodiment, the cationic polymers P) contain units of N-vinylamines of the formula (I) as polymerized units, where R.sup.1 is hydrogen or alkyl and R.sup.2 is hydrogen. Such polymers are obtainable, for example, by complete or partial hydrolysis of homo- or copolymers of open-chain N-vinylcarboxamides of the formula (V) ##STR5##

Brief Summary Text (98):

Copolymers of from 1 to 99, preferably from 30 to 70, mol % of acrylamide and/or methacrylamide and from 99 to 1, preferably 70 to 30, mol % of dialkylaminoalkyl acrylates and/or methacrylates, for example copolymers of acrylamide and N,N-dimethylaminoethyl acrylate or N,N-diethylaminoethyl acrylate, are preferred. Basic acrylates are preferably present in a form neutralized with acids or in quaternized form. The quaternization may be effected as described above. The cationic polymers have K values of from about 30 to 300, preferably from 100 to 180 (determined

according to H. Fikentscher in 5% strength aqueous sodium chloride solution, at 25.degree. C. and at a polymer concentration of 0.5% by weight).

CLAIMS:

19. A process as claimed in claim 1, wherein said anionically modified starch A) is reacted with a cationic polymer P), which comprises

i) 1 to 100 mol % of an N-vinylamine of formula I ##STR7##

wherein R.sup.1 and R.sup.2 are independently hydrogen or alkyl, and

ii) 0 to 99 mol % of a monomer selected from vinylformiate, vinylacetate, vinylpropionate, N-vinylformamide, vinylalcohol, acrylonitrile and N-vinylpyrrolidone

in the polymerized form.

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L18: Entry 1 of 1

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May 22, 2001

US-PAT-NO: 6235835

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TITLE: Polymer-modified anionic starch, method for its production, and its use

DATE-ISSUED: May 22, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Niessner; Manfred	Schifferstadt			DE
Nilz; Claudia	Rodersheim-Gronau			DE
Lorenzak; Primoz	Ludwigshafen			DE
Rubenacker; Martin	Altrip			DE
Ettl; Roland	Hassloch			DE

US-CL-CURRENT: 525/54.24; 162/168.1, 162/168.2, 162/168.3, 162/168.4, 162/175,  
525/54.26

Full	Title	Citation	Front	Review	Classification	Date	Reference	Patents	Assignments	Claims	KMC	Draw. D.
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L20: Entry 1 of 2

File: USPT

Sep 5, 2000

DOCUMENT-IDENTIFIER: US 6113785 A

TITLE: Polysulfone membrane for purifying bloodAbstract Text (1):

A polysulfone membrane for purifying blood having excellent compatibility with blood and the process producing the membrane are disclosed. The membrane comprises a mixed polymer phase of a graft copolymer and/or block copolymer having a molecular weight of 3.times.10.<sup>5</sup> daltons or more and comprising (A) a hydrophilic segment and (B) a hydrophobic segment (exclusive of polysulfone) in a total amount of from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone, with the monomer unit ratio (A/B) between the segments A and B being from 0.5 to 5. The copolymer is preferably a graft copolymer where the hydrophilic segment is a polyvinylpyrrolidone segment and the hydrophobic segment is a polystyrene segment. The membrane can be prepared by applying a wet film formation process to a dope containing an appropriate solvent of the mixed polymer, such as N,N-dimethylacetamide. The membrane for purifying blood reduces cumbersomeness in the washing of the coagulated membrane during the film formation process and allows recovery of the solvent from the coagulating solution at a high recovery rate because the copolymer contained in the structure of the membrane is substantially not eluted into water contacted during the film formation process and the washing step.

Brief Summary Text (2):

The present invention relates to a membrane for blood purification such as hemodialysis and hemofiltration and more specifically, the present invention relates to a polysulfone membrane for purifying blood having excellent compatibility with the blood and the membrane of which the membrane producing performance is improved in the washability of the membrane and recoverability of the solvent used in producing the membrane.

Brief Summary Text (4):

Polysulfone resin has been extensively applied and developed as a medical material because of its excellent heat resistance, chemical resistance and .gamma.-ray resistance. The polysulfone resin is also used as a material in highly transmissive artificial dialyzers. However, the polysulfone itself is hydrophobic and exhibits poor blood compatibility by itself. Hitherto, various methods have been developed in an attempt to improve the compatibility with the blood. For example, Japanese Unexamined Patent Publication (Kokai) No. 61-93801 discloses a method of adding polyvinylpyrrolidone to thereby improve the blood compatibility of the membrane and Japanese Unexamined Patent Publication (Kokai) No. 6-165926 discloses a polysulfone hollow fiber membrane containing a vinylpyrrolidone-base polymer and a polyglycol.

Brief Summary Text (5):

The compatibility with the blood can be improved by blending a hydrophilic polymer as in these techniques, however, since the hydrophilic polymer blended with the polysulfone resin is water-soluble, a thorough washing of the membrane formed is essential. Due to this, the washing step generally takes a long time and the film formation process is inefficient. Further, when a water-soluble hydrophilic polymer is added, in addition to the problem of a cumbersome washing process, there is a serious problem in the production, due to the fact that the water-soluble polymer

added during the film formation is eluted in a large amount into the coagulating solution. More specifically, at the time of recovering a solvent of the membrane from the coagulating solution, the solvent becomes difficult to recover because the viscosity of the coagulating solution is greatly increased due to the presence of the hydrophilic polymer.

Brief Summary Text (6):

From the standpoint of suppressing elution of the added hydrophilic polymer, for example, Japanese Unexamined Patent Publication (Kokai) Nos. 63-97205 and 4-300636 disclose a technique of subjecting a polysulfone-base membrane having added thereto a hydrophilic polymer such as polyvinylpyrrolidone, to heat treatment or radiation treatment. However, the heat treatment must be performed at a fairly high temperature (170.degree. C. or more) and the membrane performance is difficult to maintain. Further, the method of effecting cross-linking by high-power gamma-ray irradiation or the like may reduce the blood compatibility of the membrane. Furthermore, these methods cannot overcome the problem accompanying the elution of the hydrophilic polymer into the coagulating solution.

Brief Summary Text (7):

In order to improve the water permeability of the polysulfone membrane, a hydrophilic polymer having low solubility in water may be added. In this respect, a method for forming a membrane comprising adding a graft copolymer or block copolymer consisting of a polysulfone segment and a hydrophilic polymer segment is disclosed, for example, in Japanese Unexamined Patent Publication (Kokai) Nos. 62-168503, 62-199621, 62-201603, 63-88003, 63-84603 and 2-140234.

Brief Summary Text (9):

An object of the present invention is to provide a polysulfone-base hemocatharsis membrane having excellent compatibility with the blood.

Brief Summary Text (10):

Another object of the present invention is to provide a process for producing a polysulfone membrane for purification of blood having excellent compatibility with the blood through a simple washing step with a high recovery of the solvent used for the dope of the formed membrane.

Brief Summary Text (12):

The present invention has been accomplished by taking advantage of the fact that a polysulfone membrane containing a graft copolymer and/or block copolymer consisting of a hydrophilic segment and a hydrophobic segment exhibits excellent compatibility with the blood and the graft copolymer and/or block copolymer is not easily eluted into the coagulating solution at the time of forming the membrane.

Brief Summary Text (13):

More specifically, the objects of the present invention have been attained by a polysulfone membrane for purifying blood comprising a graft copolymer and/or block copolymer consisting of (A) a hydrophilic segment and (B) a hydrophobic segment (exclusive of polysulfone), the monomer unit ratio (A/B) of A to B being from 0.5 to 5 and the total of A and B being from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone.

Brief Summary Text (14):

The monomer unit A or B as used herein means a repeating unit in a polymer constituting the hydrophilic segment or the hydrophobic segment, respectively. For example, in a graft or block copolymer consisting of a polyvinylpyrrolidone segment and a polystyrene segment, the monomer units A and B are a repeating unit represented by the following formulae [I] and [II], respectively: ##STR1##

Brief Summary Text (15):

By analysis of the membrane surface, it was found that an improved eluting property

and excellent compatibility with blood of the polysulfone membrane for purifying blood of the present invention are attained by making the hydrophobic segment embedded in the polysulfone membrane, or bonded thereto by a bonding force of the affinity using a graft and/or block copolymer comprising the hydrophilic and the hydrophobic segments, whereby the ratio of the hydrophobic segment to the hydrophilic segment on the membrane surface becomes smaller than the ratio of the hydrophobic segment to the hydrophilic segment in the entire membrane.

Brief Summary Text (16):

More specifically, the present invention further provides a polysulfone membrane for purifying blood comprising a graft copolymer and/or block copolymer consisting of (A) a hydrophilic segment and (B) a hydrophobic

Brief Summary Text (17):

segment (exclusive of polysulfone), the monomer unit ratio (A/B) of A to B being from 0.5 to 5, the total of A and B being from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone, and the monomer unit ratio (U=B'/A') between the hydrophobic segment (B') and the hydrophilic segment (A') present on the surface of the membrane being smaller than the monomer unit ratio (V=B/A) between the hydrophobic segment (B) and the hydrophilic segment (A) present in the entire membrane.

Brief Summary Text (18):

The monomer unit ratio "U" as used herein is defined as a value derived from abundance ratios of the hydrophilic unit in the copolymer, the hydrophobic unit in the copolymer and the polysulfone, obtained by determining the quantities of characteristic elements of both units and polysulfone, elements in the characteristic chemical bonding state (if desired, quantity determined by the peak split process) and the constituent elements according to the ESCA (electron spectroscopy for chemical analysis). For example, a method for determining the monomer unit ratio "U" in the case of a membrane of polysulfone resin comprising a repeating unit represented by the following formula [IV] containing a copolymer consisting of a polyvinylpyrrolidone segment and a polystyrene segment is described. Abundance ratios of nitrogen originated from the vinylpyrrolidone unit and sulfur originated from polysulfone are determined by ESCA. Similarly, the abundance ratio of nitrogen in the polyvinylpyrrolidone film and the abundance ratio of sulfur in the additive-free polysulfone membrane are determined by ESCA. From the abundance ratios determined, the covering ratio of the vinylpyrrolidone unit and the exposure ratio of polysulfone are determined. Then, from the covering ratio of the vinylpyrrolidone unit and the exposure ratio of polysulfone, the covering ratio of the styrene unit is determined. From the covering ratio of the vinylpyrrolidone unit and the covering ratio of the styrene unit, the monomer unit ratio "U" of the membrane is obtained.

Brief Summary Text (19):

The polysulfone as used in the present invention is a polyaryl ether sulfone polymer characterized by the structure containing a repeating unit represented by the following formula [III]. Examples thereof include a polymer comprising a repeating unit represented by formula [IV] and a polymer comprising a repeating unit represented by formula [III]. ##STR2##

Brief Summary Text (20):

The graft copolymer and/or block copolymer consisting of (A) a hydrophilic segment and (B) a hydrophobic segment (exclusive of polysulfone) as used in the present invention means a block copolymer having a form of A--B, A--B--A, B--A--B, (A--B).sub.X --A, B--(A--B).sub.X, a graft copolymer comprising a main chain of (A) a hydrophilic segment and a branch of (B) a hydrophobic segment, or a graft copolymer comprising a trunk of (B) a hydrophobic segment and a branch of (A) a hydrophilic segment.

Brief Summary Text (21):

The copolymer preferably has a molecular weight of from 3.times.10.sup.4 to 2.times.10.sup.6 daltons.

Brief Summary Text (22):

Examples of the hydrophilic segment of the present invention include a segment comprising a polymer or copolymer of a monomer such as methacrylic acid, acrylic acid, itaconic acid, 2-hydroxyethyl methacrylate, 2-hydroxyethyl acrylate, 2-hydroxypropyl methacrylate, 2-hydroxypropyl acrylate, glycerol methacrylate, polyethylene glycol methacrylate, N,N'-dimethylacrylamide, N-methylacrylamide, dimethylaminoethyl methacrylate, methylenebisacrylamide, diacetone acrylamide, N-vinylpyrrolidone or vinyl alcohol, or a polymer such as a polyethylene glycol segment or a polypropylene glycol segment.

Brief Summary Text (23):

Examples of the hydrophobic segment of the present invention include a segment comprising a polymer or copolymer of a methacrylic ester or acrylic ester monomer such as methyl methacrylate, ethyl methacrylate, n-propyl methacrylate, n-butyl methacrylate or benzyl methacrylate, a styrene monomer such as styrene, methylstyrene or ethylstyrene, a vinyl carboxylate monomer such as vinyl acetate, or an acrylonitrile monomer.

Brief Summary Text (24):

The copolymer may be polymerized by a commonly known method. For example, a block copolymer may be synthesized from a hydrophilic monomer and a hydrophobic monomer by anionic living polymerization, cationic living polymerization or photoiniferter polymerization (see Nippon Gomu Kyokaishi (Journal of Japan Rubber Association), Vol. 59, No. 12, p. 658 (1986)). The synthesis method of the graft copolymer is described, for example, in Japanese Unexamined Patent Publication (Kokai) No. 50-77526 and Angew Makromol. Chem., Vol. 132, 81 (1985). Several Synthesis examples are described below in greater detail. A synthesis example of a block-type copolymer by the photoiniferter polymerization is described below. A hydrophilic monomer or a hydrophobic monomer and a living photopolymerization initiator having a dithiocarbamate group (e.g., benzyl N,N-diethyldithiocarbamate, p-xylenebis(N,N-diethyldithiocarbamate)) is dissolved in a solvent and polymerized by irradiating with UV light to synthesize a polymer having a growing terminal. From this reaction solution, the polymer having a growing terminal is purification-separated. This polymer and a hydrophobic monomer in the case of a polymer obtained from a hydrophilic monomer, or a hydrophilic monomer in the case of a polymer obtained from a hydrophobic monomer, are dissolved in a solvent and polymerized starting from the growing terminal by again irradiating with UV light to obtain a block copolymer. A block copolymer having a repeating unit of (A--B).sub.X --A or (B--A).sub.X --B can be obtained by repeating the purification-separation of the polymer having a growing terminal and the polymerization with a monomer under irradiation with UV light.

Brief Summary Text (25):

An example of a synthesis of a block-type copolymer by anionic living polymerization is described below. A dehydrated hydrophilic monomer or hydrophobic monomer is polymerized with a polymerization initiator (e.g., sodium naphthalene) in a dehydrated solvent to synthesize a polymer having a growing terminal. After the reaction of the monomer is completed, a dehydrated hydrophobic monomer in the case of a polymer obtained from a hydrophilic monomer, or a dehydrated hydrophilic monomer in the case of a polymer obtained from a hydrophobic monomer, is added to the reaction solution obtained above to effect polymerization starting from the growing terminal. As a result, a block copolymer is obtained. A block copolymer having a repeating unit of (A--B).sub.X --A or (B--A).sub.X --B can be obtained by repeating the addition of a monomer after completion of the monomer reaction.

Brief Summary Text (26):

A synthesis example of a graft-type copolymer by the copolymerization of a macromonomer and a monomer is described below. Macromonomers such as polyethylene glycol or polystyrene having a double bond at one terminal are commercially available but the macromonomer can be synthesized by the following method, if desired. A hydrophilic monomer or hydrophobic monomer is polymerized using azobisisobutyro-nitrile (AIBN) as a polymerization initiator and 3-mercaptopropionic acid as a chain transfer agent to synthesize a prepolymer having a carboxyl group at one terminal. The prepolymer obtained is reacted with glycidyl methacrylate and, as a result, a hydrophilic macromer or hydrophobic macromer is obtained. Other than this, the macromonomer may also be obtained by a method of polymerizing a hydrophilic monomer or hydrophobic monomer by anionic polymerization and adding methacrylic acid chloride thereto to react with the polymer to obtain a hydrophilic macromer or hydrophobic macromer having a double bond at one terminal.

Brief Summary Text (27):

A hydrophilic macromer and a hydrophobic monomer or a hydrophobic macromer and a hydrophilic monomer are polymerized in the presence of a polymerization initiator and a graft copolymer consisting of a hydrophobic segment and a hydrophilic segment is obtained.

Brief Summary Text (28):

The polysulfone-base hemocatharsis membrane of the present invention can be prepared by a so-called wet film formation process where polysulfone and a graft copolymer and/or block copolymer comprising (A) a hydrophilic segment and (B) a hydrophobic segment in a total amount of from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone, with the monomer unit ratio (A/B) between the segments A and B being from 0.5 to 5, is dissolved in a predetermined solvent to have a polysulfone concentration of from 12 to 25 wt % based on the prepared dope, the thus-prepared dope for forming a membrane is formed into a plane membrane or a hollow fiber, the plane membrane or hollow fiber formed is contacted with a predetermined coagulating solution, the solvent is removed and the residue is washed.

Brief Summary Text (29):

The polysulfone membrane for purifying blood of the present invention is advantageous in that the membrane having the above-described structure is formed during the coagulation process and the graft copolymer and/or block copolymer is substantially not eluted into the coagulating solution or the washing solution after the coagulation. Accordingly, in recovering a solvent of the membrane from the coagulating solution by distillation or the like, an increase in the viscosity ascribable to elution of the polymer does not occur and a high recovery of the solvent can be achieved. Further, since the polymer is not eluted during washing after the coagulation, the washing step can be completed within a short time.

Brief Summary Text (31):

In the graft copolymer and/or block copolymer for use in the present invention, the hydrophilic segment (A) is preferably a polyvinylpyrrolidone segment or a polyethylene glycol segment, more preferably a polyvinylpyrrolidone segment. The hydrophobic segment (B) is preferably a polymethyl methacrylate segment or a polystyrene segment, more preferably a polystyrene segment.

Brief Summary Text (32):

A graft copolymer is preferably used. The graft copolymer is preferably a graft copolymer having a trunk of polymethyl methacrylate segment or polystyrene segment and a branch of polyethylene glycol segment, or a graft copolymer having a trunk of polyvinylpyrrolidone segment and a branch of polymethyl methacrylate segment or polystyrene segment, and most preferably a graft polymer having a trunk of polyvinylpyrrolidone and a branch of polystyrene segment.

Brief Summary Text (33):

These graft copolymers and/or block copolymers can be easily prepared by a known synthesis method described in detail above.

Brief Summary Text (34):

The copolymer preferably has a molecular weight of from  $3 \times 10^4$  to  $2 \times 10^6$  daltons, more preferably from  $5 \times 10^4$  to  $1.5 \times 10^6$  daltons, still more preferably from  $1 \times 10^5$  to  $1 \times 10^6$  daltons. If the molecular weight is too small, satisfactory effects cannot be obtained by addition or the problem of cumbersomeness in the water washing step still remains, whereas if the molecular weight is too large, mixing with the polysulfone resin proceeds poorly and a uniform membrane cannot be obtained in practice. The molecular weight as used herein means a molecular weight at peak out of the molecular weight in terms of styrene obtained by the gel permeation chromatography (GPC). More specifically, it is a GPC molecular weight at peak in terms of styrene, obtained using a Shodex (trademark) GPC KD-800 series as a column, N,N-dimethylformamide containing 0.01 mol/l of lithium bromide as an eluent and a differential refractometer as a detector.

Brief Summary Text (35):

The ratio of the hydrophilic segment (A) to the hydrophobic segment (B) of the present invention is, in terms of the monomer unit ratio (A/B) of A to B, from 0.5 to 5, preferably from 1 to 4, more preferably from 1.2 to 3. The ratio of the hydrophilic segment to the hydrophobic segment is chosen so as to strike a balance between the insolubility in water and the improvement in the blood compatibility of the polysulfone-base membrane. More specifically, in the case where the ratio of the hydrophilic segment to the hydrophobic segment is too large, the problem of cumbersomeness in the water washing step is not overcome or the elution into the coagulating solution cannot be sufficiently suppressed, as a result, the recovery of the solvent can be difficult. On the other hand, if the ratio of the hydrophilic segment to the hydrophobic segment is too small, the blood compatibility of the polysulfone membrane cannot be sufficiently improved.

Brief Summary Text (36):

The hydrophilic segment (A) and the hydrophobic segment (B) each must be contained in the polysulfone-base membrane of the present invention in an amount such that the total amount of A and B is from 0.5 to 30 parts by weight, preferably from 3 to 25 parts by weight, more preferably from 6 to 20 parts by weight, per 100 parts by weight of polysulfone. If the amount of the hydrophilic segment and the hydrophobic segment contained in the membrane is too large, there arises a problem in the heat resistance or the mechanical strength of the membrane. On the other hand, if the content of these segments are too small, good compatibility with the blood cannot be attained.

Brief Summary Text (37):

The polysulfone membrane for purifying blood of the present invention preferably has a form such that in the cross-sectional structure, the monomer unit ratio ( $U=B'/A'$ ) between the hydrophobic segment (B') and the hydrophilic segment (A') present on the membrane surface is smaller than the monomer unit ratio ( $V=B/A$ ) between the hydrophobic segment (B) and the hydrophilic segment (A) present in the entire membrane. For example, when a value (W, decrement of the hydrophobic segment on the membrane surface) obtained in such a manner that the monomer unit ratio ( $U=B'/A'$ ) between the hydrophobic segment (B') and the hydrophilic segment (A') present on the membrane surface is subtracted from the monomer unit ratio ( $V=B/A$ ) between the hydrophobic segment (B) and the hydrophilic segment (A) present in the entire membrane and the result is divided by the monomer unit ratio ( $V=B/A$ ) between the hydrophobic segment (B) and the hydrophilic segment (A) present in the entire membrane, is used as an index, the index value is preferably from 0.3 to 1, more preferably from 0.5 to 1, still more preferably from 0.7 to 1.

Brief Summary Text (38):

The polysulfone-base hemocatharsis membrane of the present invention can be obtained by a wet film formation process which is a conventionally and commonly known technique. Either a so-called hollow fiber membrane having a hollow fiber form or a plane membrane may be used. The dope (stock solution for forming a membrane) for use in the wet film formation is a solution obtained by dissolving and mixing the polysulfone and the copolymer described above in a solvent which dissolves both the polysulfone and the copolymer. The solvent is not particularly limited, however, solvents such as N,N-dimethylacetamide, N,N-dimethylformamide, N-ethyl-pyrrolidone and dimethylsulfoxide have a high solubility and are easily available and therefore, these may be conveniently used. Among these solvents, N,N-dimethylacetamide is most preferred in view of solubility for polysulfone, safety to the organism and cost. These solvents may of course be used individually but may be used in combination of two or more solvents so as to adjust the solubility for the polymer.

Brief Summary Text (39):

With respect to the concentration of polysulfone, if it is too small, the membrane can be formed only with difficulty and the strength of the membrane may be lowered, whereas if it is too large, the spinning property may be worsened or the hole size may be reduced. Accordingly, the concentration of polysulfone is preferably from 12 to 25 wt %, more preferably from 15 to 20 wt %, still more preferably from 16 to 18 wt %, based on the dope. However, the concentration of polysulfone is not limited to this range and a concentration lower or higher than this range may be used so as to obtain a membrane having desired properties.

Brief Summary Text (40):

The copolymer added to the dope is scarcely eluted into the coagulation bath during the film formation and therefore, the eluted amount need not be taken into consideration. The copolymer may be added in an amount corresponding to the amount of the copolymer intended to be present in the membrane.

Brief Summary Text (41):

The plane membrane can be obtained by casting the above-described dope on a substrate such as glass plate by means of a surgical blade and then dipping it in a coagulation bath. The hollow fiber membrane can be obtained by extruding the dope from the sheath part of a spinneret of tube-in-orifice type, extruding at the same time an inner coagulating solution from the core part and, after traveling in air, dipping the fibers in a coagulation bath. The inner coagulating solution and the coagulation bath solution for use in the film formation each comprise mainly a solvent, such as water or an alcohol, in which polysulfone and the copolymer are only slightly soluble. However, in order to obtain the described properties of the hollow fiber membrane, a mixed solution of a solvent of polysulfone and the copolymer with water or an alcohol may be used. The plane membrane or hollow fiber after dipping in a coagulation bath is, if desired, further washed with water in a water washing bath. The solvent remaining in the membrane after the wash may be removed by

Brief Summary Text (42):

washing the membrane with hot water or the like. Thereafter, the membrane may be dried after attaching thereto a hole size retaining agent such as glycerine, if desired.

Brief Summary Text (43):

The amount of the hydrophilic segment or the hydrophobic segment contained in the polysulfone-base membrane may be analyzed by NMR (nuclear magnetic resonance spectroscopic method), for example, from a proton NMR spectrum obtained using a solvent capable of dissolving or thoroughly swelling the membrane. For example, in the case of a polysulfone membrane comprising a repeating unit represented by formula [IV] containing a copolymer consisting of a polyvinylpyrrolidone segment and a polystyrene segment, in the case where chloroform-d<sub>1</sub> is used as a

solvent in the analysis, the quantities of polysulfone, polyvinylpyrrolidone segment and polystyrene segment can be relatively determined from the peaks in the spectrum when the chemical shift is in the vicinity of 7.85 (4 protons), 3.2 (2 protons) and/or 3.7 (1 proton), and 6.55 (2 protons), respectively. These quantities each can be converted into the part by weight using the formula weight per unit. In the case where the system is complicated and difficult to analyze, fractions divided by the gel permeation chromatography (GPC) or liquid chromatography (LC) may be analyzed one after another by NMR.

Brief Summary Text (44) :

In analyzing the composition of the dope for forming a membrane, low molecular weight substances such as a solvent are removed by evaporation or the like, the polymer obtained is ground into a powder and in the case of containing the hydrophilic polymer and/or hydrophobic homopolymer, these are removed by washing or reprecipitation, and then the residue is dried to obtain a sample polymer composition. The finally obtained polymer composition can be analyzed as described above using NMR. After fractionating the polymer composition by GPC or LC the composition may also be analyzed by subjecting each fraction to NMR analysis or quantitative analysis.

Detailed Description Text (3) :

(1) Quantitative Evaluation of Platelets Adhered (plane membrane)

Detailed Description Text (4) :

Whole blood was sampled from a healthy man using a syringe previously containing 3.8 wt % of a sodium citrate solution (1 to 9 volume ratio of blood) and a platelet-rich plasma was prepared by centrifugal separation. Heparin (final concentration: 10 U/ml) and a calcium chloride solution (final concentration: 5 mM) were added to the plasma and the resulting plasma was contacted with a plane membrane and allowed to stand at 37.degree. C. for 1 hour. Thereafter, the membrane was washed with phosphoric acid-buffered physiological saline and then, a phosphoric acid-buffered physiological saline solution containing 0.5% of tritonX-100 was added to dissolve the adhered platelets. The lactic acid dehydrogenase activity in the solution obtained was measured by an LDH measurement kit (manufactured by Boelinger Manheim) and the change (.DELTA.ABS) in the absorbance was quantitatively evaluated. The value obtained and converted into a unit area equivalence (unit: IU/m.sup.2) was used as an index for the quantity of platelet adhered.

Detailed Description Text (8) :

The coagulation bath solution used in the formation of a membrane and the hot water used in the washing of the membrane were combined and subjected to superfractionation under reduced pressure using a fractionating column until the viscosity of the solution amount to 500 pma.cndot.s (measuring temperature: 25.degree. C.) to recover the solvent used in the dope and the coagulating solution. From the amount of solvent recovered and the amount of solvent used (sum of the amount of the solvent in the dope used and the amount of the solvent in the coagulating solution used), the solvent recovery (%) was obtained.

Detailed Description Text (9) :

(4) Quantitative Determination of Additives in Membrane

Detailed Description Text (10) :

The membrane was thoroughly dried and then dissolved in chloroform-d.sub.1 and the NMR of the solution obtained was measured. The quantities of polysulfone, polyvinylpyrrolidone segment and polystyrene segment were relatively determined from the peaks in the spectrum when the chemical shift was in the vicinity of 7.85 (4 protons), 3.2 and 3.7 (3 protons in total), and 6.55 (2 protons), respectively. These quantities each was converted into the part by weight using the formula weight per unit. The monomer unit ratio A/B of (A) polyvinylpyrrolidone segment to

(B) polystyrene segment was obtained by the relative determination values.

Detailed Description Text (11):

(5) Quantitative Determination of Membrane Surface Composition

Detailed Description Text (12):

The membrane was thoroughly dried and the abundance ratios of nitrogen originated from vinylpyrrolidone unit and sulfur originated from polysulfone polymer on the membrane surface were determined by ESCA (electron spectroscopy for chemical analysis). Similarly, the abundance ratio of nitrogen in the polyvinylpyrrolidone membrane and the abundance ratio of sulfur in the additive-free polysulfone membrane were determined by ESCA. From the abundance ratios determined, the covering ratio (X %) of the vinylpyrrolidone unit and the exposure ratio (Y %) of polysulfone on the membrane surface were determined according to the following equations (1) and (2).

Detailed Description Text (13):

(6) Decrement of Hydrophobic Segment on the Membrane Surface

Detailed Description Text (14):

From the exposure ratio (Y %) of polysulfone and the covering ratio (X %) of vinylpyrrolidone unit obtained by the method described in the evaluation method (5), the monomer unit ratio ( $U=B'/A'$ ) between the styrene unit ( $B'$ ) and the vinylpyrrolidone unit ( $A'$ ) present on the membrane surface was determined according to the following equation (3). Further, the monomer unit ratio ( $V=B/A$ ) between the styrene unit ( $B$ ) and the vinylpyrrolidone unit ( $A$ ) present in the entire membrane was determined by the method described in the evaluation method (4). And, according to the following equation (4), a value obtained by subtracting the monomer unit ratio ( $U=B'/A'$ ) between the styrene unit ( $B'$ ) and the vinylpyrrolidone unit ( $A'$ ) present on the membrane surface from the monomer unit ratio ( $V=B/A$ ) between the styrene unit ( $B$ ) and the vinylpyrrolidone unit ( $A$ ) present in the entire membrane was divided by the monomer unit ratio ( $V=B/A$ ) between the styrene unit ( $B$ ) and the vinylpyrrolidone unit ( $A$ ) present in the entire membrane to obtain a value ( $W$ , decrement of the hydrophobic segment on the membrane surface).

Detailed Description Text (16):

15 parts by weight of styrene polymer (AS-6, produced by Toa Gosei KK, GPC peak top molecular weight: 14,000 daltons), 85 parts by weight of N-vinylpyrrolidone, 0.2 parts by weight of azobisisobutyronitrile and 100 parts by weight of N,N dimethylacetamide were charged into a brown bottle and dissolution mixed while stirring with a mixing rotor. The mixed solution obtained was transferred into a glass-made ampule bottle and the ampule was freeze deaerated in a vacuum and then the ampule was sealed.

Detailed Description Text (17):

The mixed solution in the ampule was heated in a constant-temperature water bath at 60.degree. C. for 8 hours to effect polymerization. From the resulting solution, the solvent was removed under heating in a vacuum and the solid matter obtained was ground into fine powder. The fine powder obtained was washed with cyclohexane in a Soxhlet's extractor, dried and then washed with methanol in a Soxhlet's extractor to remove unreacted styrene macromer, by-produced polyvinylpyrrolidone and the like by extraction. The residue was thoroughly dried in a vacuum drier to obtain a graft polymer as white fine powder. The graft polymer obtained had a monomer unit ratio ( $A/B$ ) of (A) polyvinylpyrrolidone segment to (B) polystyrene segment of 2.3 and a GPC molecular weight at peak of 2.1.times.10.sup.5 daltons. Subsequently, a dope comprising 18 parts by weight of polysulfone (UDEL P-1700 (trademark), produced by Teijin Acomo Engineering Plastics KK) comprising a repeating unit represented by formula [VI] and 79 parts by weight of N,N-dimethylacetamide per 3 parts by weight of the graft copolymer obtained above was prepared. The dope obtained was cast on a glass plate by means of a surgical blade, dipped in a water bath adjusted at a

temperature of 40.degree. C. to effect phase separation and then washed three times with hot water at 70.degree. C. for 1 hour by exchanging the hot water. As a result, Plane Membrane A was obtained. Plane Membrane A was subjected to the quantitative evaluation of platelets adhered and the result is shown in Table 1 as a relative value to the lactic acid dehydrogenase activity per the membrane unit area, which is an index for the quantity of platelets adhered to the polysulfone membrane, in Comparative Example 1 taken as 100. The amount of the graft copolymer remaining in the membrane and calculated from the graft copolymer content of the plane membrane determined by the NMR analysis and the amount of the graft copolymer added to the dope, is also shown in Table 1 together with the graft copolymer composition (A/B) in the membrane. Further, the membrane surface composition (the exposure ratio of polysulfone polymer and the covering ratio of vinylpyrrolidone unit) determined by the ESCA is shown in Table 6 and the decrement of the hydrophobic segment on the membrane surface determined by the ESCA and NMR analysis is shown in Table 7.

Detailed Description Text (19) :

Plane Membrane B was manufactured in the same manner as in Example 1 except for using a dope composition comprising 1 part by weight of the graft copolymer, 18 parts by weight of polysulfone and 81 parts by weight of N,N-dimethylacetamide. Plane Membrane B was evaluated on the quantity of platelet adhered, the residual amount of graft copolymer, the graft copolymer composition in the membrane, the membrane surface composition and the decrement of the hydrophobic segment on the membrane surface in the same as in Example 1. The results obtained are shown in Tables 1, 6 and 7.

Detailed Description Text (21) :

Plane Membrane C was manufactured in the same manner as in Example 1 except for not using the graft copolymer and using a dope composition comprising 18 parts by weight of polysulfone and 82 parts by weight of N,N-dimethylacetamide. Plane Membrane C was evaluated on the quantity of platelet adhered, and the lactic acid dehydrogenase activity per the unit membrane area obtained was taken as 100.

Detailed Description Text (23) :

Plane Membrane D was manufactured in the same manner as in Example 2 except for using a dope composition comprising 1 part by weight of polyvinylpyrrolidone (K-90, produced by ISP Japan KK), 18 parts by weight of polysulfone and 81 parts by weight of N,N-dimethylacetamide. The quantity of platelet adhered was evaluated in the same manner as in Example 1 and the amount of polyvinylpyrrolidone remaining in the membrane was evaluated by NMR in the same manner as in Example 1. The results are shown in Table 1.

Detailed Description Text (25) :

Plane Membrane E was manufactured in the same manner as in Example 2 except for using a dope composition comprising 5 parts by weight of polyvinylpyrrolidone (K-90), 18 parts by weight of polysulfone and 77 parts by weight of N,N-dimethylacetamide. The quantity of platelets adhered was evaluated in the same manner as in Example 1 and the amount of polyvinylpyrrolidone remaining in the membrane was measured by NMR in the same manner as in Example 1. The results are shown in Table 1.

Detailed Description Text (27) :

A graft copolymer was prepared in the same manner as in Example 1 except for using 13 parts by weight of styrene macromonomer, .87 parts by weight of N-vinylpyrrolidone and 0.15 parts by weight of azobisisobutyronitrile. The graft copolymer obtained had a monomer unit ratio (A/B) of (A) polyvinylpyrrolidone segment to (B) polystyrene segment of 2.5 and a GPC molecular weight at peak of 3.8.times.10.sup.5 daltons. Subsequently, a hollow fiber was spun, using this graft copolymer, as follows. A dope comprising 18 parts by weight of polysulfone (UDEL P-1700) and 79 parts by weight of N,N-dimethylacetamide per 3 parts by weight of the

graft copolymer obtained above was prepared. The dope was spun into a hollow fiber having an inner diameter of 200 .mu.m and an outer diameter of 290 .mu.m using a 40% N,N-dimethylacetamide aqueous solution as the inner coagulating solution and water as the outer coagulating solution. Hollow Fiber A sampled immediately after taking it out from the coagulation bath was washed eight times with hot water at 90.degree. C. for 20 minutes. A hollow fiber was sampled after every washing and the amount of graft copolymer remaining in the hollow fiber was determined by NMR in the same manner as in Example 1. The result obtained is shown in Table 2.

Detailed Description Text (30):

A hollow fiber was spun in the same manner as in Example 3 except for using a dope comprising 5 parts by weight of polyvinylpyrrolidone (K-90), 18 parts by weight of polysulfone and 77 parts by weight of N,N-dimethylacetamide. Hollow Fiber B sampled immediately after taking it out from the coagulation bath was determined on the amount of polyvinylpyrrolidone remaining in the hollow fiber at the washing process in the same manner as in Example 3. The result is shown in Table 2.

Detailed Description Text (33):

A hollow fiber (Hollow Fiber A') after hot water washing (90.degree. C., 20 minutes, 8 times) of Hollow Fiber A obtained in Example 3 was evaluated on the quantity of platelets adhered. A relative value to the lactic acid dehydrogenase activity per the unit area, which is an index for the quantity of platelets adhered to the polysulfone hollow fiber, in Comparative Example 5 taken as 100 is shown in Table 4.

Detailed Description Text (35):

A hollow fiber (Hollow Fiber C) was spun in the same manner as in Example 3 except for using a dope comprising 1 part by weight of polyvinylpyrrolidone (K-90), 18 parts by weight of polysulfone and 81 parts by weight of N,N-dimethylacetamide. Hollow Fiber C after hot water washing (90.degree. C., 20 minutes, 8 times) was evaluated on the quantity of platelets adhered and the lactic acid dehydrogenase activity per the unit area on the inner surface of Hollow Fiber C was taken as 100.

Detailed Description Text (37):

A hollow fiber (Hollow Fiber D) was spun in the same manner as in Example 3 except for using a dope comprising 1 part by weight of the graft copolymer, 18 parts by weight of polysulfone and 81 parts by weight of

Detailed Description Text (38):

N,N-dimethylacetamide. Hollow Fiber D after hot water washing (90.degree. C., 20 minutes, 8 times) was evaluated on the quantity of platelet adhered. A relative value to the lactic acid dehydrogenase activity per the unit area, which is an index for the quantity of platelet adhered to the polysulfone hollow fiber, in Comparative Example 5 taken as 100 is shown in Table 4.

Detailed Description Text (40):

A graft copolymer was prepared in the same manner as in Example 1 except for using 20 parts by weight of styrene macromonomer, 80 parts by weight of N-vinylpyrrolidone and 0.10 parts by weight of azobisisobutyronitrile. The graft copolymer obtained had a monomer unit ratio (A/B) of (A) polyvinylpyrrolidone segment to (B) polystyrene segment of 1.8 and a GPC molecular weight at peak of 7.9.times.10.sup.5 daltons. Subsequently, a dope comprising 18 parts by weight of polysulfone (UDEL P-1700) and 79 parts by weight of N,N-dimethylacetamide per 3 parts by weight of the graft copolymer obtained above was prepared. The dope obtained was cast on a glass plate by means of a surgical blade, dipped in a water bath adjusted at a temperature of 40.degree. C. to effect phase separation and then washed three times with hot water at 70.degree. C. for 1 hour by exchanging the hot water. As a result, Plane Membrane F was obtained. Plane Membrane F was subjected to the quantitative evaluation of platelets adhered. A relative value of the lactic

acid dehydrogenase activity per the membrane unit area, which is an index for the quantity of platelet adhered to the polysulfone membrane, in Comparative Example 6 taken as 100, and the graft copolymer content (parts by weight) of the plane membrane per 100 parts by weight of polysulfone determined by the NMR analysis are shown in Table 5.

Detailed Description Text (42):

Plane Membrane G was manufactured in the same manner as in Example 6 except for using a dope comprising 1 part by weight of the graft copolymer, 18 parts by weight of polysulfone and 81 parts by weight of N,N-dimethylacetamide. The lactic acid dehydrogenase activity and the graft copolymer content determined in the same manner as in Example 6 are shown in Table 5.

Detailed Description Text (44):

Plane Membrane H was manufactured in the same manner as in Example 6 except for using a dope comprising 0.5 parts by weight of the graft copolymer, 18 parts by weight of polysulfone and 81.5 parts by weight of N,N-dimethylacetamide. The lactic acid dehydrogenase activity and the graft copolymer content determined in the same manner as in Example 6 are shown in Table 5.

Detailed Description Text (46):

Plane Membrane I was manufactured in the same manner as in Example 6 except for using a dope comprising 0.2 parts by weight of graft copolymer, 18 parts by weight of polysulfone and 81.8 parts by weight of N,N-dimethylacetamide. The lactic acid dehydrogenase activity and the graft copolymer content determined in the same manner as in Example 6 are shown in Table 5.

Detailed Description Text (48):

100 parts of dehydrated styrene and 500 parts of dehydrated tetrahydrofuran were charged in a flask and, thereto, 1.4 parts of 1.6 mol/l. cndot.n-butyl lithium was added while keeping the temperature at -20.degree. C. with stirring in a nitrogen atmosphere. After continuing stirring for 8 hours, the reaction solution was transferred to a flask containing dry ice and stirred. Thereafter, the solution was poured into a large amount of a methanol solution of hydrochloric acid and then a white precipitate was obtained. The precipitate was separated by filtration, washed with water and dried under heating in a vacuum to obtain a styrene polymer having a carboxyl terminal group as white solid matter. 40 parts by weight of the white solid matter obtained, 60 parts by weight of polyvinylpyrrolidone (K-60, ISP Japan KK), 0.2 parts by weight of dimethylaminopyridine and 400 parts by weight of purified chloroform were placed in a flask and dissolution-mixed under stirring. Thereto, 0.25 parts by weight of dicyclohexyl-carbodiimide was added and the mixed solution was stirred for 6 hours. From the resulting solution, the solvent was removed under heating in a vacuum and then a white solid matter was obtained. This solid matter was grained into fine powder. The fine powder obtained was washed with toluene in a Soxhlet's extractor, dried and then washed with methanol in a Soxhlet's extractor. Thereafter, the fine powder was thoroughly dried in a vacuum drier to obtain a block copolymer as white fine powder. The block copolymer obtained had a monomer unit ratio (A/B) of (A) polyvinylpyrrolidone segment to (B) polystyrene segment of 2.2 and a GPC peak top molecular weight of 270,000 daltons. Subsequently, Plane Membrane J was manufactured in the same manner as in Example 6 except for using a dope comprising 18 parts by weight of polysulfone (UDEL P-1700) and 81 parts by weight of N,N-dimethylacetamide per 1 part by weight of the block copolymer obtained above. The lactic acid dehydrogenase activity and the block copolymer content determined in the same manner as in Example 6 are shown in Table 5.

Detailed Description Text (50):

Plane Membrane C obtained in Comparative Example 1 was evaluated on the quantity of platelet adhered simultaneously with the plane membranes obtained in Example 6 to 9. The lactic acid dehydrogenase activity per the unit membrane area determined for

Plane Membrane C was taken as 100.

Detailed Description Text (52):

A graft copolymer was prepared in the same manner as in Example 1 except for using 40 parts by weight of styrene polymer, 60 parts by weight of N-vinylpyrrolidone and 0.25 parts by weight of azobisisobutyronitrile. The graft copolymer obtained had a monomer unit ratio (A/B) of (A) polyvinylpyrrolidone segment to (B) polystyrene segment of 0.9 and a GPC molecular weight at peak of  $2.5 \times 10^5$  daltons. Subsequently, a dope comprising 18 parts by weight of polysulfone (UDEL P-1700) and 81.5 parts by weight of N,N-dimethylacetamide per 0.5 parts by weight of the graft copolymer obtained above was prepared. The dope obtained was cast on a glass plate by means of a surgical blade, dipped in a water bath adjusted at a temperature of 40.degree. C. to effect phase separation and then washed three times with hot water at 70.degree. C. for 1 hour by exchanging the hot water. As a result, Plane Membrane K was obtained. Plane Membrane K was evaluated on the membrane surface composition and the decrement of the hydrophobic segment on the membrane surface in the same manner as in Example 1. The results are shown in Tables 6 and 7.

Detailed Description Text (54):

An N-vinylpyrrolidone-styrene random copolymer emulsion (ANTARA430, produced by ISP) was concentrated and dried to solidify. As a result, N-vinylpyrrolidone-styrene random copolymer was obtained. Then, Plane Membrane L was manufactured in the same manner as in Example 2 except for using a dope comprising 18 parts by weight of polysulfone (UDEL P-1700) and 81 parts by weight of N,N-dimethylacetamide per 1 part by weight of the random copolymer obtained above. Plane Membrane L was evaluated on the membrane surface composition and the decrement of the hydrophobic segment on the membrane surface in the same manner as in Example 1. The results are shown in Tables 6 and 7.

Detailed Description Text (58):

The amount of graft copolymer and the amount of block copolymer are the parts by weight of the graft copolymer and the parts by weight of the block copolymer, respectively, when the amount of polysulfone in the plane membrane is taken as 100 parts by weight.

Detailed Description Text (60):

The polysulfone membrane for blood purification of the present invention has excellent compatibility with the blood in methods and is useful as a membrane for purifying blood such as hemodialysis and hemofiltration.

Detailed Description Text (61):

The polysulfone membrane for purifying blood of the present invention uses a hydrophobic graft copolymer and/or block copolymer containing an appropriate amount of a hydrophobic component within the molecule as an additive compatible with the blood and therefore, the additive has strong affinity for the polysulfone resin and is not easily eluted even when the membrane is contacted with washing water, water for priming or the like.

Detailed Description Text (62):

Due to this, the membrane of the present invention can be produced by a process simplified in the water washing step and elevated in the solvent recovery efficiency. Further, the membrane can be produced by a process which dispenses with the work for preventing elution of the polymer, such as cross-linking treatment.

Detailed Description Paragraph Equation (1):

Covering ratio (X %) of vinylpyrrolidone unit = (abundance ratio of nitrogen on the membrane surface/abundance ratio of nitrogen in the polyvinylpyrrolidone film)  $\times 100\%$  Equation (1):

Detailed Description Paragraph Equation (2):

Exposure ratio (Y %) of polysulfone=(abundance ratio of sulfur on the membrane surface/abundance ratio of sulfur in the additive-free polysulfone membrane).times.100(%) Equation (2):

Detailed Description Paragraph Table (1):

TABLE 1	Ratio of Quantity of Copolymer
Platelets Remaining in Membrane Adhered Membrane (%) A/B	Example 1 Plane 23 .+- . 6 96 2.3 Membrane A
Example 2 Plane 36 .+- . 16 97 2.4 Membrane B Comparative Plane 100 .+- . 29 -- --	
Example 1 Membrane C Comparative Plane 51 .+- . 19 48 -- Example 2 Membrane D	
Comparative Plane 27 .+- . 12 45 -- Example 3 Membrane E	

Detailed Description Paragraph Table (2):

TABLE 2	Ratio of Copolymer Remaining in
Membrane (%) Number of Times Example 3 Comparative Example 4 of Washing (Hollow Fiber A) (Hollow Fiber B)	Example 1 Plane 23 .+- . 6 96 2.3 Membrane A
97 47 3 96 45 4 98 44 5 96 46 6 96 45 7 97 45 8 96 45	0 96 62 1 97 51 2

Detailed Description Paragraph Table (5):

TABLE 5	Quantity of Amount of Graft
Platelets Copolymer or Amount Membrane Adhered of Block Copolymer	Example 6 Plane 10 .+- . 3 16 parts by weight
Membrane F Example 7 Plane 27 .+- . 8 5.3 parts by weight Membrane G Example 8 Plane 56 .+- . 16 2.7 parts by weight Membrane H Example 9 Plane 50 .+- . 3 1.1 parts by weight Membrane I Example 10 Plane 31 .+- . 7 5.4 parts by weight Membrane J	Example 1 Plane 23 .+- . 6 96 2.3 Membrane A
Comparative Plane 100 .+- . 9 0 part by weight Example 10 Membrane C	0 96 62 1 97 51 2

Detailed Description Paragraph Table (6):

TABLE 6	Covering Ratio (X) of
Vinylpyrrolidone Exposure Ratio (Y) Unit (%) of Polysulfone (%)	Example 1 43.2 56.3 Example 2 34.5 64.3
Example 11 18.2 73.7 Comparative 15.5 49.0 Example 7	

Detailed Description Paragraph Table (7):

TABLE 7	Decrement of Hydrophobic Segment on
the Membrane Surface	Example 1 0.97 Example
2 0.92 Example 11 0.60 Comparative Example 7 0.02	

Current US Original Classification (1):

210/500.41

CLAIMS:

1. A polysulfone membrane for purifying blood comprising a polysulfone and a graft copolymer consisting of (A) a hydrophilic segment and (B) a hydrophobic segment, the monomer unit ratio (A/B) of (A) to (B) being from 0.5 to 5 and the total of (A) and (B) being from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone, wherein (B) is not polysulfone, and wherein (A) the hydrophilic segment and (B) the hydrophobic segment form the graft copolymer.
2. The polysulfone membrane for purifying blood as claimed in claim 1, wherein (A) a hydrophilic segment and (B) a hydrophobic segment form the block copolymer.
3. A polysulfone membrane for purifying blood comprising a polysulfone and a graft copolymer and/or block copolymer consisting of (A) a hydrophilic segment and (B) a

hydrophobic segment, the monomer unit ratio (A/B) of (A) to (B) being from 0.5 to 5 and the total of (A) and (B) being from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone, wherein (B) is not polysulfone, and wherein (B) the hydrophobic segment is a polystyrene segment.

4. The polysulfone membrane for purifying blood as claimed in claim 3, wherein the hydrophilic segment is a polyvinylpyrrolidone segment.

5. A polysulfone membrane for purifying blood comprising a polysulfone and a graft copolymer and/or block copolymer consisting of (A) a hydrophilic segment and (B) a hydrophobic segment, the monomer unit ratio (A/B) of (A) to (B) being from 0.5 to 5 and wherein the total of the hydrophilic segment (A) and the hydrophobic segment (B) is from 6 to 20 parts by weight per 100 parts by weight of polysulfone, wherein (B) is not polysulfone.

6. The polysulfone membrane for purifying blood as claimed in claim 5, wherein the monomer unit ratio (A/B) of the hydrophilic segment (A) to the hydrophobic segment (B) is from 1 to 4.

7. The polysulfone membrane for purifying blood as claimed in claim 5, wherein the monomer unit ratio (A/B) of the hydrophilic segment (A) to the hydrophobic segment (B) is from 1.2 to 3.

8. A process for producing a polysulfone membrane for purifying blood, comprising dissolving polysulfone and a graft copolymer comprising (A) a hydrophilic segment and (B) a hydrophobic segment in a total amount of from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone, with the monomer unit ratio (A/B) between the segments (A) and (B) being from 0.5 to 5, in a predetermined solvent to have a polysulfone concentration of from 12 to 25 wt % based on the dope prepared, forming the film formation dope into a plane membrane or a hollow fiber, contacting the plane membrane or hollow fiber formed with a predetermined coagulating solution, removing the solvent and washing the residue; wherein (B) is not polysulfone, and (A) the hydrophilic segment and (B) the hydrophobic segment form the graft copolymer.

9. The process for producing a polysulfone membrane for purifying blood as claimed in claim 8, wherein the hydrophilic segment is a polyvinylpyrrolidone segment and the hydrophobic segment is a polystyrene segment.

10. A polysulfone membrane for purifying blood comprising a polysulfone and a graft copolymer and/or block copolymer consisting of (A) a hydrophilic segment and (B) a hydrophobic segment, the monomer unit ratio (A/B) of (A) to (B) being from 0.5 to 5 and the total of (A) and (B) being from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone, wherein (B) is not a polysulfone, and wherein the monomer unit ratio ( $U=B'/A'$ ) between the hydrophobic segment (B') and the hydrophilic segment (A') present on the surface of the membrane is smaller than the monomer unit ratio ( $V=B/A$ ) between the hydrophobic segment (B) and the hydrophilic segment (A) present in the entire membrane.

11. The polysulfone membrane for purifying blood as claimed in claim 10, wherein the value (W) obtained by dividing a value resulting from subtracting the monomer unit ratio ( $U=B'/A'$ ) between the hydrophobic segment (B') and the hydrophilic segment (A') present on the membrane surface from the monomer unit ratio ( $V=B/A$ ) between the hydrophobic segment (B) and the hydrophilic segment (A) present in the entire membrane, by the monomer unit ratio ( $V=B/A$ ) between the hydrophobic segment (B) and the hydrophilic segment (A) present in the entire membrane is from 0.3 to 1.

12. A process for producing a polysulfone membrane for purifying blood comprising dissolving polysulfone and a graft copolymer and/or block copolymer comprising (A)

a hydrophilic segment and (B) a hydrophobic segment in a total amount of from 0.5 to 30 parts by weight per 100 parts by weight of polysulfone, with the monomer unit ratio (A/B) between the segments (A) and (B) being from 0.5 to 5, in a predetermined solvent to have a polysulfone at a concentration of from 12 to 25 wt % based on the dope prepared, forming the film formation dope into a plane membrane or a hollow fiber, contacting the plane membrane or hollow fiber formed with a predetermined coagulating solution, removing the solvent and washing the residue; wherein (B) is not polysulfone, and (B) the hydrophobic segment is a polystyrene segment.

13. The process for producing a polysulfone membrane for purifying blood as claimed in claim 12, wherein (A) a hydrophilic segment and (B) a hydrophobic segment form the block copolymer.

14. The process for producing a polysulfone membrane for purifying blood as claimed in claim 12, the hydrophilic segment is a polyvinylpyrrolidone segment.

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\*\* See image for Certificate of Correction \*\*TITLE: Water-absorbing, essentially water-free membrane for reagent substrates and methods of preparing the sameAbstract Text (1):

In a reagent strip for the dry-chemical detection of a component of an aqueous sample solution comprising a detection reagent in a membrane, the improvement wherein the membrane is an essentially anhydrous, but water-absorbing, membrane for a reagent substrate in an analytical agent for the dry-chemical detection of a component of an aqueous sample solution, comprising at least one hydrophobic film-forming, water insoluble organic polymer in which at least one hydrophilic organic polymer is dispersed as discrete particles. The reagent can be incorporated into the membrane when produced or can later be applied as by impregnation. The membrane performs as well as or better than multi-layer strips.

Brief Summary Text (1):

The invention relates to novel membranes and their use as reagent substrates for analytical agents which can be employed for the dry-chemical determination of components of aqueous solutions, mainly body fluids, such as blood, urine, serum, milk or the like, and their preparation from a water-in-oil emulsion of a hydrophilic water-containing, and preferably water-soluble, polymer and a solution of a hydrophobic, water-insoluble, film-forming polymer.

Brief Summary Text (5):

A frequently used material for test strips is paper, which is impregnated with reagent solutions and employed directly or in conjunction with polymeric carrier materials or additional components, such as hydrophobic polymers or semipermeable membranes. Such test agents are described, for example, in U.S. Patent Specification Nos. 3,092,463 and 3,092,465, and EP-A 78 971. However, fibrous materials, such as paper, have serious disadvantages as reagent substrates in test strips. These disadvantages are described in detail in U.S. Patent Specification No. 4,042,335. Here, it is intended only to discuss the poorly reproducible analytical values, which are due to the non-uniformity of the paper material and at best permit a semiquantitative interpretation of the results.

Brief Summary Text (27):

The invention relates to a water-absorbing, essentially water-free membrane for reagent substrates in analytical agents for the dry-chemical detection of components of aqueous sample solutions, which is characterized in that it comprises one or more hydrophobic, film-forming, water-insoluble organic polymers in which one or more hydrophilic, preferably water-soluble, organic polymers are distributed as a discontinuous phase.

Brief Summary Text (28):

Preferably, the hydrophilic polymers are contained in the pores of the membrane, the coherent phase of which consists of the film-forming, water-insoluble polymers.

Brief Summary Text (29):

The membranes according to the invention can be obtained by mixing a solution of one or more film-forming, water-insoluble organic polymers with a dispersion of one or more hydrophilic, water-containing polymers in an organic solvent, applying the mixture onto a base and drying it.

Brief Summary Text (30):

In another method for the preparation of the membranes according to the invention, the hydrophilic, water-containing polymer is dispersed in a solution of the film-forming, water-insoluble polymer in an organic solvent, and this dispersion, which corresponds to the above mixture in its composition, is applied onto a base and dried.

Brief Summary Text (32):

Reagent substrates according to the invention consist of one of the membranes defined above, which contains one or more detection reagents (preferably all those required) for the determination of a component of the sample.

Brief Summary Text (35):

In the preparation of the reagent substrates according to the invention, the detection reagents are either dissolved in the mixture from which the membrane is prepared, or added in the form of a water-in-oil (W/O) dispersion or (less preferably) distributed in the membrane by subsequent impregnation of the latter. The organic solvents which may be used in this procedure should satisfy the criteria stated further below.

Brief Summary Text (36):

The preferably solid base for membrane production can consist of glass, metal or a synthetic or natural polymer which, when the mixture is applied and dried in order to produce the membrane or reagent substrate, is not completely dissolved or not surface-swollen to such an extent that it changes its external form. The base can be transparent or opaque. It preferably consists of polyethylene terephthalate, to which pigments can be added in order to render it in transparent to light, or of polyethylene-coated paper.

Brief Summary Text (37):

The membrane or the reagent substrate can also be produced on one of the abovementioned bases, detached from this, and applied, by means of a suitable adhesive, onto another base, which in this case can also consist of a material, such as polycarbonate, cellulose acetate, polystyrene etc., which is soluble in customary organic solvents.

Brief Summary Text (38):

Suitable film-forming, water-insoluble organic polymers are those which absorb less than 1% by weight of water by swelling, which have a solubility of more than 1% by weight in one of the organic solvents described further below, and which give a film after the solvent has been evaporated. The polymers can be, for example, polymers of ethylenically unsaturated monomers, polyaddition or polycondensation products, or natural products or derivatives of these. Soluble polyhydrocarbons, polydienes, polyvinyl compounds, polyacrylates or -methacrylates, polycarbonates, polyacetals, polyethers, polyurethanes, phenol resins, polyesters, polysulphones, polyamides and polyimides and organo-soluble polysaccharides and derivatives of these are preferably used.

Brief Summary Text (39):

Particularly preferred among these are: bisphenol A polycarbonate, polystyrene, poly-p-methylstyrene, polytrifluoromethylstyrenes, polymethyl methacrylate, cellulose acetate, cellulose acetobutyrate, cellulose acetopropionate, styrene/acrylonitrile copolymers, styrene/maleic anhydride copolymers, methyl

methacrylate/methyl acrylate copolymers, aromatic polyesters, tetramethylbisphenol A polycarbonate, polyvinyl acetate, polyisobutylene, copolymers of styrene and butadiene or isoprene and copolymers of acrylonitrile and butadiene, the monomer building of which in each case can be arranged randomly or blockwise, and ethylcellulose.

Brief Summary Text (40):

Of course, it is also possible to use mixtures of the abovementioned polymers, or copolymers having a random or blockwise distribution of the monomer units.

Brief Summary Text (48):

The hydrophilic polymers which are employed according to the invention can be of natural or synthetic origin and ionic or non-ionic. For example, starch, gelatine or derivatives thereof, agar-agar, pullulanes, hydrophilic cellulose derivatives and hydrophilic polymers and copolymers can be used. Among these, the following may be mentioned: polyethyleneimine, polyacrylic and methacrylic derivatives, such as polyacrylamide, poly-N-methyl- or N,N-dimethylacrylamide, polyvinyl alcohol, polyacrylic acid and salts thereof, polyalkylaminoalkyl methacrylates or -amides and salts of these, polystyrenesulphonic acid and salts thereof, polyacrylamido-2-methyl-2-propanesulphonic acid and salts thereof, polyvinylimidazole, poly-N-vinylactams, such as poly-N-vinylpyrrolidone or polyvinylcaprolactam, poly-N-vinylamides, such as poly-N-Vinyl-N-methylacetamide, poly-N-vinylurethanes, copolymers with malic anhydride and copolymers which contain basic building blocks of the above polymers distributed randomly or blockwise.

Brief Summary Text (50):

The amount of the detergents should be chosen so that a fine dispersion having sufficient stability for the duration of the process for the preparation of the membrane is formed.

Brief Summary Text (55):

After the dispersing process, the dispersion can be diluted or concentrated again in order to match its viscosity to, for example, the viscosity of the solution of the hydrophobic polymer in an organic solvent, with which solution the dispersion is mixed in order to produce the membrane. This mixing process can be carried out either by vigorous shaking or with the aid of mechanical apparatuses, such as stirrers, mixing nozzles, etc. For predetermined concentrations of the two phases, the volume ratio of the W/O dispersion of the hydrophilic, water-containing polymers to the solution of the water-insoluble, film-forming polymer depends on the amounts of hydrophilic and water-insoluble, film-forming polymers which are required for functioning of the membrane and are described further below. If a certain volume ratio is prescribed for technical reasons, the ratio of the amounts of film-forming and hydrophilic polymers in the membrane must be set via their concentrations in the phases used for membrane production.

Brief Summary Text (57):

To produce the membrane, the mixture described above is applied onto a substrate, and this can be achieved by painting, spraying, applying with a knife-coater or casting. Uniform thicknesses can best be applied using appropriate mechanical apparatuses which are familiar to the skilled worker. Since the viscosity of the mixture can be varied within wide limits, optimum casting behavior can be established for any casting technique. The thickness of the freshly applied layer is preferably between 10 .mu.m and 1,000 .mu.m and depends on the solids content of the mixture and the desired thickness of the finished membrane. This is obtained by drying the mixture applied on the substrate. Drying can be accelerated by blowing on heated air, by heating or by applying a vacuum. In certain circumstances, low-boiling solvents evaporate sufficiently rapidly at room temperature even without additional measures.

Brief Summary Text (59):

Drying may also be carried out in a plurality of stages, drying in the 1st stage advantageously being carried out using air which has a high moisture content. In the subsequent stages, it is possible to use air having lower moisture contents and, for example, higher temperatures. The final drying stage should be carried out using heated air (temperature between 20.degree. and 80.degree. C., preferably between 30.degree. and 60.degree. C.) which has a low moisture content, if complete drying of the membrane is desired.

Brief Summary Text (60):

In a preferred embodiment of the invention, the dispersion of the hydrophilic, water-containing polymer in the organic solvent (oil phase) is prepared by inverse emulsion polymerization (water-in-oil) of an aqueous solution of monomers or monomer mixtures which is dispersed in the oil phase. The inverse emulsion polymerization process is known, and is described, for example, in U.S. Patent Specification Nos. 3,284,393 and 3,691,124, DE-OS (German Published Specification) Nos. 1,745,176, 2,432,699 and 2,926,103, EP-A 68 955 and British Patent Specification No. 1,482,515. Emulsifiers, polymeric stabilizers, initiators, oil phases and possible monomers and monomer combinations which can advantageously be employed are also listed there. W/O dispersions which are particularly suitable for the preparation of the membranes or reagent substrates according to the invention are obtained in inverse emulsion polymerization of the following neutral, basic or acidic monomers, or combinations thereof: acrylamide, methacrylamide, N,N-dimethylacrylamide, N-vinylpyrrolidone, N-vinylcaprolactam, N-vinyl-N-methylacetamide, N-vinylacetamide, N-vinylurethanes; acrylic acid, methacrylic acid, styrenesulphonic acid, acrylamido-2-methyl-2-propanesulphonic acid and salts of these, N-vinylimidazole, dimethylaminoethyl methacrylate and -methacrylamide, dimethylaminopropyl methacrylate and -methacrylamide, and salts thereof, as well as mixtures of these monomers. Very particularly preferred monomers are acrylamide and mixtures of the stated monomers with acrylamide.

Brief Summary Text (64):

In the preparation of the membranes or reagent substrates according to the invention, various property-modifying additives can be introduced into the mixture of the solution of the film-forming, water-insoluble organic polymers and the dispersion of the hydrophilic, water-containing polymers. To increase the reflection, pigments, such as titanium dioxide, silica gel and the like, can be dispersed in the casting mixture. Other possible additives are thickeners for increasing the viscosity, or compounds which are suitable for improving the adhesion of the reagent substrate to the base when the reagent substrate remains on the base, or to reduce the said adhesion when the reagent substrate is to be detached from the base. With the aid of surface-active substances or wetting agents, it is possible to modify the surface properties of the reagent substrate in order, for example, to improve wetting by the sample material or to increase the brilliance of the reaction color. The properties of the reagent substrate are also affected by plasticizers, for example the hardness and strength, the adhesion, etc.

Detailed Description Text (2):

In a 1 face-ground vessel equipped with a paddle stirrer, 440 g of chloroform, 12 g of RTM.Span 80 (Span 80 is a sorbitan monooleate), 4 g of a copolymer of stearyl methacrylate and acrylamide (proportions by weight 82.6/17.4) and 0.2 g of azo-bis-(isobutyronitrile) are initially taken, degassed and heated to 50.degree. C. At this temperature, 20% of a degassed solution of 40 g of acrylamide and 80 g of H.sub.2O is rapidly added dropwise under a gentle stream of nitrogen. The remainder of the monomer solution is added dropwise in the course of 2 hours. When the addition is complete, stirring is continued for 2 hours. The dispersion contains 6.9% by weight of polyacrylamide.

Detailed Description Paragraph Table (1):

TABLE 1

Example Emulsifier Monomers No. Organic phase (weight/.sup.+.) (weight/%) H.sub.2 O content.sup.++ T .degree.C.

2

trichloreoethylene Span 80 (2.6) acrylamide (6.9) 66.7 60 3 " Span 60 (2.6)  
 acrylamide (6.9) 60 70 4 toluene Span 80 (4.3) acrylamide (8.2) 66.7 55 5.sup.++++  
 cyclohexane Span 60 (10.0) acrylamide (11.2) 60 80 6 chloroform Span 80 (3.5)  
 acrylamide (6.9) 80 50 7.sup.+++ " Span 80 (3.5) acrylamide (6.9) 66.7 55 8 " Span  
 80 (2.6) acrylamide (13.2) 66.7 55 9.sup.++++ " Span 80 (2.6) acrylamide (6.9) 66.7  
 55 10 " " N,N--dimethylacrylamide (6.9) " " 11 " " N,N--dimethylaminoethyl " "  
 methacrylate.HCl/acrylamide (1.7/5.2) 12 " " Na acrylate/acrylamide (4.0/3.4) " "  
 13 " " N--vinylpyrrolidone/acrylamide (3.4/3.4) " " 14 " " methacrylamide (6.9) " "  
 15 " " N--vinylacetamide/acrylamide (3.5/3.5) " " 16 " " N--  
 vinylimidazole/acrylamide (3.5/3.5) " "

.sup.+

relative to organic phase .sup.++ relative to aqueous phase .sup.+++ without the addition of stearyl methacrylate/acrylamide copolymer .sup.++++ with 0.5 g of azobis (isobutyronitrile)

Detailed Description Paragraph Table (3):

1 Copolymer of 80% of styrene and 20% of acrylonitrile; 2 Polymethyl methacrylate; [.eta.] (25.degree. C., tetrahydrofuran) = 0.6 3 Copolymer of 80% of styrene and 20% of maleic anhydride; 4 Copolymer of 75% of styrene, 15% of acrylonitrile and 10% of maleic anhydride; 5 Copolymer of 56% of styrene, 28% of methyl methacrylate and 16% of maleic anhydride; 6 Cellulose acetate; .eta..sub.rel (20% strength in acetone: ethanol = 9:1) = 180 7 Cellulose acetobutyrate; .eta..sub.rel (20% strength in acetone:ethanol = 9:1) = 200 8 Cellulose acetopropionate; .eta..sub.rel (20% strength in acetone:ethanol = 9:1) = 200 9 Polycarbonate obtained from tetramethylbisphenol A; .eta..sub.rel (measured on a 0.5% strength solution in methylene chloride) = 1.33 10 Polyisobutylene; (.sup.R Oppanol B 100) 11 Polyester obtained from tetramethyl-bisphenol A and terephthalic acid; .eta..sub.rel (measured on a 0.5% strength solution in methylene chloride) = 1.4 12 Polystyrene; [.eta.] = 0.9 (toluene, 25.degree. C.) 13 Polyvinyl acetate; molecular weight: 37,000 g/mol 14 Polycarbonate of bisphenol A; .eta..sub.rel , measured on a 0.5% strength solution in methylene chloride = 1.28 15 Polycarbonate of bisphenol A; .eta..sub.rel (0.5% strength in methylene chloride) = 1.29 16 Polycarbonate of bisphenol A, with 0.5 mol % of isatin-biscresol as branching agent; .eta..sub.rel (0.5% strength in methylene chloride) = 1.32 17 Polycarbonate of bisphenol A; .eta..sub.rel , measured on a 0.5% strength solution in methylene chloride = 1.30 18 Poly-p-methylstyrene; [.eta.] = 0.8 (toluene, 25.degree. C.) 19 SBS 3-block copolymer consisting of 30% of styrene and 70% of butadiene, prepared by anionic poly- merisation in hexane using butyl-lithium as an initiator, molecular weight: 150,000 g/mol.

CLAIMS:

1. An essentially anhydrous, but water-absorbing, membrane for a reagent substrate in an analytical agent for the dry-chemical detection of a component of an aqueous sample solution, comprising at least one hydrophobic film-forming, water-insoluble organic polymer selected from the group consisting of polyhydrocarbons, polydienes, polyvinyl compounds, polyacrylates or -methacrylates, polycarbonates, polyacetals, polyethers, polyurethanes, phenol resins, polyesters, polysulphones, polyamides and polyimides and organo-soluble polysaccharides in which at least one hydrophilic organic polymer capable of absorbing at least 20% by weight of water is dispersed as discrete particles.
2. The membrane according to claim 1, wherein the at least one hydrophilic polymer is contained in pores of a coherent phase of the membrane made up of the at least one hydrophobic, water-insoluble polymer.

3. The membrane according to claim 1, wherein the weight ratio of the at least one hydrophilic polymer to the at least one hydrophobic polymer is between 10:1 and 1:10.

4. A process for producing an essentially anhydrous, but water-absorbing, membrane for a reagent substrate in an analytical agent for the dry-chemical detection of a component of an aqueous sample solution, comprising mixing a solution of at least one film-forming, hydrophobic water-insoluble organic polymer selected from the group consisting of polyhydrocarbons, polydienes, polyvinyl compounds, polyacrylates or -methacrylates, polycarbonates, polyacetals, polyethers, polyurethanes, phenol resins, polyesters, polysulphones, polyamides and polyimides and organo-soluble polysaccharides with a dispersion of at least one hydrophilic, water-containing organic polymer, wherein the hydrophilic polymer is capable of absorbing at least 20% by weight of water, in an organic solvent to form a mixture, applying the mixture onto a base, and drying.

5. A process for producing an essentially anhydrous, but water-absorbing, membrane for a reagent substrate in an analytical agent for the dry-chemical detection of a component of an aqueous sample solution, comprising dispersing at least one hydrophilic, water-containing, organic polymer, wherein the hydrophilic polymer is capable of absorbing at least 20% by weight of water, in a solution of at least one film-forming, hydrophobic, water-insoluble organic polymer selected from the group consisting of polyhydrocarbons, polydienes, polyvinyl compounds, polyacrylates or -methacrylates, polycarbonates, polyacetals, polyethers, polyurethanes, phenol resins, polyesters, polysulphones, polyamides and polyimides and organo-soluble polysaccharides to form a dispersion, applying the dispersion onto a base, and drying.

6. In a reagent strip for the dry-chemical detection of a component of an aqueous sample solution comprising a detection reagent in a membrane, the improvement wherein the membrane is a membrane according to claim 1.

8. A process for preparing a reagent strip which includes a detection reagent in a membrane comprising mixing a solution of at least one film-forming, water-insoluble organic polymer selected from the group consisting of polyhydrocarbons, polydienes, polyvinyl compounds, polyacrylates or -methacrylates, polycarbonates, polyacetals, polyethers, polyurethanes, phenol resins, polyesters, polysulphones, polyamides and polyimides and organo-soluble polysaccharides with at least one hydrophilic organic polymer which contains at least 20% by weight of water to form a mixture, with the solution of the at least one water-insoluble polymer or the at least one hydrophilic polymer or both being pre-mixed with a reagent, applying the mixture onto a base, and drying.

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TITLE: Water-absorbing, essentially water-free membrane for reagent substrates and methods of preparing the same

DATE-ISSUED: October 25, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Piejko; Karl-Erwin	Cologne			DE
Bomer; Bruno	Bergisch-Gladbach			DE
Bartl; Herbert	Odenthal			DE
Frank; Georg	Elkhart	IN		

US-CL-CURRENT: 422/56; 422/57, 427/384, 428/327, 435/805, 436/169

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## Article



## Separation Science and Technology

**Publisher:** Taylor & Francis**Issue:** Volume 38, Number 3 / 2003**Pages:** 591 - 612**URL:** [Linking Options](#)**DOI:** 10.1081/SS-120016653**Microfiltration of Activated Sludge Using Modified PVC Membranes: Effect of Pulsing on Flux Recovery**Dae Sik Kim <sup>A1</sup>, Jong Seok Kang <sup>A1</sup>, Young Moo Lee <sup>A1</sup><sup>A1</sup> National Research Laboratory for Membrane, School of Chemical Engineering, College of Engineering, Hanyang University, Seongdong-KU, Seoul, South Korea**Abstract:**

A combined effect of backpulsing, crosspulsing, and membrane-surface modification was investigated for the reduction of membrane fouling and the recovery of flux for crossflow filtration. The hydrophobic poly(vinyl chloride) (PVC) membranes were subjected to the surface modification with N-vinyl-2-pyrrolidinone (NVP) monomer by the ultraviolet-assisted graft polymerization to increase the surface wettability and to decrease the adsorptive fouling. The flux was dependent on the membrane-surface properties. The flux of the modified membrane was higher than that of the unmodified membrane due to the increase of hydrophilicity on the membrane surface and the more or less dilution of protein concentration in the sludge solution at NVP layer. The recovered fluxes of modified and unmodified membranes, after the removal of the foulants from the membrane surface, were about 61% and 34% of the initial flux, respectively. These results suggest that the adhesive interactions of the mixed liquor-suspended solid (MLSS) with the hydrophilic membrane surfaces are weaker than those with a hydrophobic surface. To clean foulants,

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backpulsing and crosspulsing were carried out. 3.6-fold and 5.6-fold enhancements of modified membranes in the average permeate flux, in comparison with unmodified membranes, were obtained for filtering MLSS using backpulsing alone and a combination of backpulsing and surface modification, respectively. The average permeate flux of surface modified membrane with backpulsing (5.6-fold) was higher than with crosspulsing (2.8-fold), indicating that the deposition of MLSS in the interior of the membrane pore was the dominant fouling mechanism. The transmembrane pressure (TMP) of modified membranes was lower than that of unmodified membrane.

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**Keywords:**

Microporous membrane, Poly(vinylchloride), N-vinyl-2-pyrrolidinone (NVP), Sludge, Crossflow filtration, Backpulsing

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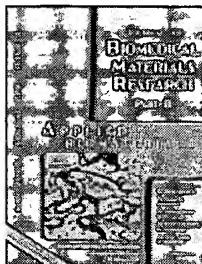
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**Properties and biocompatibility of polypropylene graft copolymer films**

H. A. Abd El-Rehim <sup>\*</sup>, M. B. El-Arناouty

National Centre for Radiation Research and Technology, Atomic Energy Authority, P.O. Box 29, Nasr City, Cairo, Egypt

email: H. A. Abd El-Rehim ([ha\\_rehim@hotmail.com](mailto:ha_rehim@hotmail.com))

\*Correspondence to H. A. Abd El-Rehim, National Centre for Radiation Research and Technology, Atomic Energy Authority, P.O. Box 29, Nasr City, Cairo, Egypt

**Keywords**

radiation grafting • copolymer • polypropylene • sodium acrylate • blood compatibility

**Abstract**

Modifying the surfaces of polymers has received a great deal of attention, because it could bring about specific surface characteristics such as antithrombogenic property. Therefore, N-vinyl-pyrrolidone/sodium acrylate (NVP/Na-Ac) binary monomers were introduced onto polypropylene (PP) films by a radiation grafting method. The effect of solvent and comonomer composition on the degree of grafting was determined. Studies of the mechanical properties and water content of such graft copolymers showed that as the grafting yield increases the elongation percent decreases. However, the water content increases with increasing grafting yield. The blood compatibility of the original PP and PP-g-NVP/Na-Ac films was evaluated by determination of the extent of platelet adsorption and thrombus formation. The blood compatibility of PP-g-NVP/Na-Ac seems to be better than that of original PP. © 2003 Wiley Periodicals, Inc. *J Biomed Mater Res Part B: Appl Biomater* 68B: 209-215, 2004

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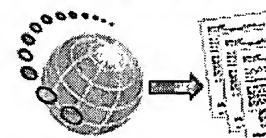
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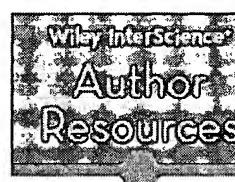
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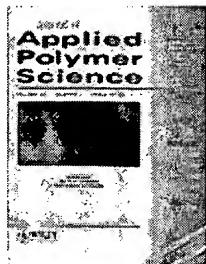
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## Development of membranes by radiation grafting of acrylamide into polyethylene films: Characterization and thermal investigations

Bhuvanesh Gupta <sup>1</sup>\*, Nishat Anjum <sup>1,2</sup>

<sup>1</sup>Department of Textile Technology, Indian Institute of Technology, New Delhi-16, India

<sup>2</sup>Department of Applied Chemistry, Delhi College of Engineering, Bawana Road, Delhi, India

email: Bhuvanesh Gupta ([bgupta@textile.iitd.ernet.in](mailto:bgupta@textile.iitd.ernet.in))

\*Correspondence to Bhuvanesh Gupta, Department of Textile Technology, Indian Institute of Technology, New Delhi-16, India

### Keywords

Polyethylene; acrylamide; membrane; radiation grafting

### Abstract

Polyethylene-g-polyacrylamide membranes were prepared by graft polymerization of acrylamide into polyethylene films by preirradiation technique. The characterization and thermal behavior of membranes with different degrees of grafting were evaluated by density, X-ray diffraction, thermogravimetric analysis, and differential scanning calorimetry measurements. Grafting led to considerable changes in the structure of polyethylene membranes. The density of the polyethylene film increased with the increase in the degree of grafting, although the increase beyond 100% grafting was less pronounced than at lower graft levels. The heat of fusion and the crystallinity of polyethylene decreased with the increase in the degree of grafting. The decrease in crystallinity is because of the cumulative effect of the dilution of inherent crystallinity by the incorporation of amorphous polyacrylamide grafts within the noncrystalline region of polyethylene (dilution effect) and partial disruption of the crystallites (crystal defects). X-ray diffraction measurements also revealed a decrease in the crystallinity in grafted films. Membranes behaved as a two-component system where polyethylene and polyacrylamide components underwent independent degradation irrespective of the graft levels. In general, the thermal stability of polyethylene in membranes was markedly improved by the grafting of acrylamide monomer as evident from the initial decomposition temperature increasing from 311°C for virgin PE to 390°C in grafted membranes. © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 82: 2629-2635, 2001

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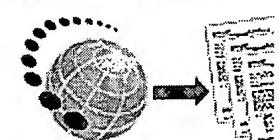


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